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RESEARCH MEMORANDUM

SURVEY OF LESS-INFLAMMABLE HYDRAULIC

FLUIDS FOR AIRCRAFT

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NATIONAL ADVISORY COMMITTEE
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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

RESEARCH MEMORANDUM

SURVEY OF LESS-INFLAMMABLE HYDRAULIC FLUIDS FOR AIRCRAFT

By Wray V. Drake and I. L. Drell

SUMMARY

Because hydraulic fluids were the initial inflammables in a number of aircraft fires, attempts have been made by private and government research organizations to produce hydraulic fluids with increased resistance to fire. A survey of the developments in this field is presented herein. Specification requirements, physical and chemical properties, hydraulic-system test results, and advantages and disadvantages of various hydraulic fluids are discussed. Types of less-inflammable hydraulic fluid reported are: glycol derivative, water base, silicone, ester, and halogenated compound. For completely satisfactory service, some modification of currently available fluids or of present hydraulic-system parts still appears necessary.

Various glycols, glycol ethers, polyglycols, and polyglycol ethers and esters have been considered as components. Least inflammability was noted for glycols of low molecular weight and for polymeric glycols and polymeric glycol ethers. Fluids with some promising properties have been devised that contain polymeric glycol ethers plus dicarboxylic or phosphate esters.

Less-inflammable hydraulic fluids have been formulated with water and ethylene glycol as main components. Improvement of low-temperature characteristics, diminished wear in pumps, and decreased attack on active metals are among the objectives sought in the continued development of this type of fluid.

Silicones of suitable viscosity exhibited many desirable qualities that recommend their use for less-inflammable hydraulic fluids. Difficulties (later partly resolved) were encountered in excessive wear due to sliding friction between steel-to-steel and steel-to-cast-iron surfaces, shrinking and hardening of O-ring rubber packings, and maintenance of tight connections in hydraulic lines.

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Of the various esters tested as components - phosphates, dicarboxylates, borates, and carbonates - the phosphates appeared the most difficult to ignite and the most stable to hydrolysis. Esters exhibited tendencies towards oxidation and corrosion and caused greater swelling of conventional rubber packings than petroleum-base hydraulic fluids.

A number of chloro and chlorofluoro organic compounds have shown promise as snuffer constituents of less-inflammable hydraulic fluids. Many fluorocarbons investigated were insufficiently miscible with other components over the appropriate temperature range. A number of fluids containing halogenated compounds caused undue swelling of present rubber packings. The toxic effect of many of these compounds is yet to be determined.

INTRODUCTION

A survey was made at the NACA Lewis laboratory of fires occurring in commercial air-carrier accidents in the United States and dependent territories during the 10-year period ending July 1, 1948 (reference 1). Of the fires in which the initial combustible was identified, 5 percent was attributed to hydraulic fluids. A more detailed analysis follows:

Type of fire	Total number of fires	Number of fires with initial combustible identified	Number of fires caused by hydraulic fluids
Ground	82	72	6
Flight	135	110	3
Crash	61	7	1

The problem of fires resulting from use of inflammable hydraulic fluids seemingly can be solved and therefore has received considerable attention during and since World War II.

The fire hazard that arises from use of inflammable hydraulic fluids is a matter of serious concern to commercial and military operators because of the wide distribution within the airplane of lines carrying the fluid under pressure. In some aircraft, these pressures are as high as 3000 pounds per square inch. Leakage of the fluid is not uncommon; in addition, a break in the line will permit a stream of fluid or a very inflammable mist to be exposed to a possible ignition source.

Many organizations have been actively engaged in the development of less-inflammable hydraulic fluids; as a result, several promising fluids have been produced. Details on the various types of fluid are presented herein, although information on a few is not given. As a participant in the program for the reduction of fire hazards in aircraft, the NACA Lewis laboratory, in reference 2, briefly presents the progress in the development of less-inflammable hydraulic fluids. Since the publication of reference 2, a more extensive survey has been made by the NACA in an effort to ascertain the current status of the development of less-inflammable fluids. The results of this survey are summarized herein in order to provide a basis for future effort in this field.

HISTORY OF HYDRAULIC-FLUID DEVELOPMENT

Water was the earliest liquid utilized as a hydraulic fluid; however, its usage was somewhat limited by poor lubricity. In the search for liquids of more desirable lubricity, subsequent development led toward liquids that are by nature highly inflammable. The first of this class was a castor-oil-base type that contained ethyl alcohol as a diluent (reference 3). This fluid was employed in the brake system of a number of automobiles in the middle 1920's (reference 4) and improved castor-oil-base formulations were later used in aircraft hydraulic systems. In recent years, petroleum-base fluids having suitable viscosities, pour points, and low-temperature stability have been used in order to permit cold-weather operation of aircraft. The importance of petroleum-base fluids has increased steadily since the early 1930's.

Early in World War II, the United States military authorities started programs to develop noninflammable or less-inflammable hydraulic fluids for aircraft. Near the close of the war, a number of airplane manufacturers, petroleum refiners, chemical companies, and associated industries became active in the development of such fluids. As a result, certain chemical and physical facts have already become well established and some fields of related research have been extensively investigated. Details of this development will be subsequently discussed.

HYDRAULIC-FLUID SPECIFICATIONS

Although inflammability characteristics must obviously serve as the basis for selection of liquids to be considered as components of less-inflammable hydraulic fluids, many other requirements must be

met before a newly developed fluid can be accepted. The chemical nature of the less-inflammable liquids considered at present is, however, such that complete conformance with specifications written for petroleum-base fluids is expected to be quite difficult.

For several years, the Bureau of Aeronautics, Department of the Navy, has had a noninflammable-hydraulic-fluid specification (reference 5) that applies to one specific water-base type. In table I, specification AN-0-366 for the conventional petroleum-base hydraulic fluid is compared with two general specifications for less-inflammable fluids, SAE AMS 3150A and a proposed military specification dated December 12, 1949. The SAE specification (reference 6) is an interim type for use in commercial-aircraft hydraulic systems; the proposed military specification, which is for developmental purposes only, presents the joint requirements of the Air Force and the Bureau of Aeronautics. Some of the specification requirements are discussed in the following paragraphs.

Packings. - Synthetic rubber packings are used to insure proper performance and tight connections for the hydraulic system. Experience has shown that constituents of even petroleum-base hydraulic fluids must be controlled to keep rubber swelling within suitable limits. A still greater problem in this regard is presented by a less-inflammable hydraulic fluid because a greater variety of types of chemical may be employed.

Compatibility. - New hydraulic fluids should be compatible with petroleum-base hydraulic fluids; the formation of immiscible layers, precipitates, or sludges would be unsatisfactory. Compatibility requirements for SAE AMS 3150A hydraulic fluids are less severe than demands made by the proposed military specification.

Toxicity. - The less-inflammable hydraulic fluids desired by both military and commercial groups should be nontoxic. Some adjustment of demands may be necessary and logical, considering the present acceptability of the use of tetraethyl lead in the fuel.

Viscosity. - Because commercial aircraft are not generally subjected to the very low temperatures military aircraft must meet, the low-temperature viscosity requirements for the less-inflammable hydraulic fluid for commercial airplanes are not as severe as are the military demands. Fluids for commercial airplanes may have viscosities at -40° F as high as 2000 or 7000 centistokes in contrast to the present 500-centistoke limit at -40° F or the roughly corresponding 2500-centipoise limit at -65° F proposed for military aircraft.

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Low-temperature stability. - Hydraulic fluids for aircraft use may be exposed to low temperatures for long periods of time. A fluid that forms solids or two liquid phases under low-temperature conditions is undesirable. Military requirements in this respect are considered to be more severe than commercial requirements.

Corrosion. - Both military and commercial requirements avoid corrosion of metallic parts of the hydraulic system. The influence of moisture is considered in testing the corrosiveness of the less-inflammable hydraulic fluid for commercial aircraft.

Oxidation. - Oxidation of fluids containing petroleum fractions produces acidic materials and sludges, particularly in the presence of certain metals. The extent of oxidation of less-inflammable hydraulic fluids is also determined and controlled.

Shear stability. - Military and commercial groups are both interested in shear stability of the fluid. Details of test conditions for each group are given in table I; conditions for the commercial fluid apparently are the most rigorous.

Wear and lubrication. - Control of these properties in the case of less-inflammable fluids for military and commercial aircraft is quite important, as noted in the specifications of table I.

Inflammability. - The term "less-inflammable" is used herein to describe a fluid that ignites with greater difficulty than a petroleum-base hydraulic fluid but that will show slight ignition in any of the inflammability tests of the SAE AMS 3150A specification; the term "noninflammable" is restricted to the condition where no flame whatever occurs in tests carefully performed in accordance with this specification. The inflammability characteristics of hydraulic fluids cannot be defined adequately by a single procedure. For this reason, several of the following methods are required in various specifications:

1. Autogenous-ignition-temperature test. - The SAE specification (reference 6) states that the autogenous-ignition temperature shall preferably be above 750° F, as determined by A.S.T.M. Method D286-30. The autogenous-ignition temperature (also called spontaneous-ignition temperature) is the temperature at which the fluid ignites upon immediate contact with a heated surface. An earlier version of the proposed military specification required an autogenous-ignition temperature of at least 1000° F; the present version, dated December 12, 1949, does not include an autogenous-ignition-temperature test.

2. Oxygen-demand test. - The oxygen-demand test was developed at the Naval Research Laboratory (reference 7) and is designed to

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measure the degree of inflammability in terms of the amount of oxygen in an oxygen-nitrogen mixture necessary to propagate an arc-ignited flame in a finely divided spray of fluid in that atmosphere. In general, substances found to be quite inflammable required as little as 12 percent oxygen in the gas mixture; these substances included, for example, benzene, n-hexadecane, and some samples of castor-oil-base and petroleum-base hydraulic fluids. In contrast, a few chlorinated substances, certain carbonate esters, some silicones, aqueous solutions of several glycols or their monoether derivatives required over 60 percent oxygen. The earlier version of the proposed military specification for less-inflammable fluids required that the percentage of oxygen needed for flame propagation be limited to a minimum of 50 percent; the present version of this specification does not include an oxygen-demand test.

3. Spray test. - In this test, the hydraulic fluid shall not increase the intensity of a standard fire when emitted in a dense spray. The SAE specification (reference 6) requires that the test fluid be equal to or better than the standard reference fluid HS-1.

4. High-temperature-ignition spray test. - The specifications require that the fluid be sprayed through an orifice under high pressure and that an attempt be made to obtain continuous ignition with a concentrated flame source. In order to pass the proposed military specification, the fluid shall stop burning upon removal of the flame source and shall not ignite except around the flame-source area itself.

5. Manifold test. - In this test, the hydraulic fluid is dripped onto a simulated manifold section heated to approximately 1300° F. The proposed military specification requires that the fluid shall not ignite on the manifold or upon leaving the manifold.

6. Incendiary-gunfire test. - The gunfire test is a military test and is not specified for the commercial type of fluid. The proposed military specification requires that the fluid shall not burn when subjected to direct penetration by a 0.30-caliber incendiary bullet. Incendiary-firing tests on several fluids of interest are described in reference 7. The height of the resulting flame was considered to be a criterion of the relative flammability of the hydraulic fluid.

DEVELOPMENT OF LESS-INFLAMMABLE HYDRAULIC FLUIDS

As previously mentioned, inflammability tests have served as the basis for the selection of potential components of less-inflammable

hydraulic fluids. Preliminary inflammability tests, conducted by the Naval Research Laboratory (references 8 and 9) and summarized in reference 7, indicated the following classes of material to be of promise:

- (1) Certain polyalkylene oxides
- (2) Glycols with high percentages of oxygen
- (3) Certain aqueous organic solutions containing sufficiently high proportions of water to render them noninflammable
- (4) Silicones with viscosities of over 20 centistokes at 100° F, if properly stripped of volatile fractions or impurities (The substances of particular interest are polymethylsiloxanes and poly(methyl, phenyl) siloxanes.)
- (5) Chlorinated or fluorinated hydrocarbons and ethers that contain approximately three atoms of halogen per molecule (For substances studied, this amount was equivalent to nearly 50-percent halogenation of the compound.)

Among types of material considered unfavorable for use as non-inflammable fluids at the time the investigation was initiated were certain organic phosphates and esters of dibasic acids (references 7 and 9). Both ester classes are discussed later because other investigators believe suitable less-inflammable fluids may be formulated by using constituents from these classes. (Two different criterions of inflammability were utilized by the respective research groups.)

On the basis of availability, hydrolytic properties, deleterious effect on packings, toxicity, and cost, classes (4) and (5) were not given immediate consideration after preliminary investigations by the Naval Research Laboratory (reference 9). Primary emphasis in the development of a new hydraulic fluid was thus placed on those chemicals in classes (1), (2), or (3). In actual practice, all three classes were involved because preliminary surveys showed that polyalkylene oxides and glycols alone did not possess requisite properties for a noninflammable fluid (references 9 and 10). Ultimately from these fundamental considerations, the Naval Research Laboratory evolved Hydrolubes C, A, U, and U-4, although other versions of Hydrolube U have received consideration.

GLYCOL DERIVATIVES

A number of glycols and glycol derivatives have been considered as possible components of less-inflammable hydraulic fluids. Most of the published information on the subject has been issued by the Naval Research Laboratory (references 7 to 11).

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Monomeric Glycols

Inflammability data for only a few of the simple alkylene glycols have been reported. Ethylene glycol is indicated (table II) to be slightly less inflammable than propylene glycol. Both were considerably less inflammable than petroleum-base hydraulic fluids and would pass the autogenous-ignition-temperature test of the SAE AMS 3150A but not the corresponding test nor the oxygen-demand test of the earlier proposed military specification. In the incendiary-gunfire test, ethylene glycol occasionally produced flames of 3 to 8 feet in height, which the investigators considered small (reference 11). Water solutions of this glycol produced flames of lower magnitude. This type of solution will be more fully discussed in the section on water-base fluids.

Higher members of the alkanediol series are available up to octanediols but little or no information has been published on inflammability and other properties of interest in hydraulic-fluid behavior.

Monomeric Glycol Ethers

Simple glycol ethers (monoalkoxyalkanols) have shown much greater inflammability than the corresponding glycols. Thus, the monomethyl, monoethyl, and monobutyl ethers of ethylene glycol had much lower flash points and oxygen demands than ethylene glycol itself, as shown in table II. These ethers were at least as inflammable as petroleum-base fluids.

In addition to low flash points, other disadvantages of lower glycols and glycol ethers as possible constituents of less-inflammable hydraulic fluids (reference 11) are: low oxidation stabilities, hygroscopic nature (causes aqueous corrosion problems), low viscosity indices, and solvent action for the plasticizers of rubber packings (particularly true for some glycol ethers of low molecular weight).

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Some of these difficulties might be overcome with the aid of viscosity-index improvers and other additives. With a few possible exceptions, the lower glycols and glycol ethers, however, are considered to be insufficiently fire resistant to show promise as less-inflammable hydraulic-fluid components unless they are used with a highly efficient snuffer or a flame-extinguishing agent such as water.

Polymeric Glycols

Such polymers as diethylene, triethylene, tetraethylene, hexaethylene, and nonaethylene glycols may now be obtained as well as homologous polypropylene and polybutylene glycols. The autogenous-ignition temperatures and oxygen demands for the first few members of the polyethylene glycol series is shown in table I of reference 10 to decrease with a higher degree of polymerization although the flash points increase. Boiling points rise with the degree of polymerization but the freezing points also increase. A commercial polyethylene glycol (predominantly nonaethylene glycol) with a molecular weight of about 400 had a flash point of 435° F but the freezing range was 4° to 10° F (reference 12).

Four points of the polyalkylene glycols could be lowered by the use of isoalkylene units to give branched-chain structures; viscosities would be increased at the same time. Nevertheless, reference 11 does not consider the polyalkylene glycols promising because of their low viscosity indices.

The low viscosity indices for the polymeric alkylene glycols are thought probably to be due to the hydrogen bonding that involves the terminal -OH groups (reference 11). This particular linkage can be reduced in amount or eliminated by placing an ester or ether group in one or both terminal positions. Association is then much less and higher viscosity indices can be expected. Such compounds are commercially available in various types and viscosity grades.

Polymeric Glycol Ethers

Two ethers of polyglycols having a low degree of polymerization are included in table II, namely, the monoethyl ether of diethylene glycol and dimethoxytetraethylene glycol. Both ethers had oxygen demands of only 12 percent, the same as that of petroleum-base fluids. However, many of the alkyl mono- and di-ethers of higher polymers of ethylene and propylene glycols, identified as HB and IB fluids, respectively, are considerably more fire resistant.

The LB and HB fluids having viscosities of over 10 centistokes at 100° F have (reference 11) flash points between 325° and 490° F; fire points between 375° and 600° F, and autogenous-ignition temperatures between 720° and 750° F. Oxygen demands range from 54 percent for more viscous grades to 20 percent for grades with viscosities below 10 centistokes at 100° F. Incendiary-gunfire tests of these fluids show results similar to those for ethylene glycol.

The LB fluids are slightly water soluble and have water tolerances of 1 to 3 percent; the HB fluids are completely miscible with water at ordinary temperatures (reference 13). Rust inhibitors are needed and various polar-type inhibitors have been developed (reference 11).

The LB and HB fluids also need oxidation inhibitors because in the pure form they oxidize in a week at 150° F; effective anti-oxidants are available for these fluids (reference 11). These polyethers are good lubricants and have little hardening or swelling effect on most rubber packings (references 13 and 14). Oxidation products are either volatile or soluble in the fluids and difficulties with sludge formation rarely occur (reference 11). The principal disadvantage of these polyethers as aircraft hydraulic fluids is probably that fluids with viscosities of 10 centistokes at 130° F have viscosities of about 10,000 centistokes at -40° F (reference 11).

Formulations

The addition of high-boiling-point organic thinners with high viscosity indices and low pour points has been suggested to overcome the aforementioned disadvantage of the polyethers (reference 11). Thus, when 40 percent by weight of the high-boiling-point aliphatic dicarboxylic ester, di-(2-ethylhexyl) adipate, was added to LB-140 fluid, the viscosity changed from 30,000 to 5500 centistokes at -40° F and the viscosity at 130° F changed from 18.1 to 10.1 centistokes. No loss in flame resistance occurred; the flash point and autogenous-ignition temperature remained the same as that for the undiluted LB-140 fluid and the oxygen demand of the blend was above 50 percent. This blend would not meet the most rigid specifications for less-inflammable hydraulic fluids but at least on viscosity properties, it satisfies the requirements of SAE AMS 3150A (grade 7000). The possibility remains that better formulations of this type, containing polyethers as primary constituents, might be developed.

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Formulations with polyethers as secondary constituents have been studied. In order to demonstrate viscosity possibilities, Carbide and Carbon Chemicals Corporation, the producer of HB and LB fluids, prepared a blend containing 27 percent by weight of HB fluid (grade 5100) and 73 percent by weight of triethyl phosphate (reference 11). This blend had viscosities of 10.3 centistokes at 130° F and 442 centistokes at -40° F, easily meeting the difficult viscosity requirements of the AN-O-366 specification. This blend was definitely more fire resistant than the AN-O-366 fluid with a flash point approximately 100° F higher, an autogenous-ignition temperature at least 300° F higher, and an oxygen demand of 55 percent. On these inflammability properties at least, this blend would pass the requirements of SAE AMS 3150A. Satisfactory wear preventatives and corrosion and rust inhibitors could probably be obtained for this fluid (reference 11); however, the large proportions of phosphate ester caused excessive swelling of the rubber packings.

Advantages and Disadvantages

On the basis of present knowledge, glycols of low molecular weight exhibit greatly decreased inflammability but are deficient in low-temperature viscosity properties; their ethers are too inflammable, have poor oxidation stability, low viscosity indices, and solvent action for rubber-packing plasticizers. The polyglycols show moderate resistance to fire and have high boiling points, but indicate the disadvantage of high freezing points. Their viscosity characteristics are poor, possibly because of associated molecules. Some promise has been shown by the ethers or esters of polyglycols in respect to viscosity and viscosity indices.

Further research in this field might include LB fluids of suitable viscosity plus halogenated esters, ethers, or hydrocarbons.

WATER-BASE FLUIDS

The Naval Research Laboratory has experimentally established the less-inflammable nature of ethylene, diethylene, and propylene glycols and their aqueous solutions, as well as aqueous solutions of certain ethers of ethylene, diethylene, and tetraethylene glycols; the results of this investigation are presented in table II. The aqueous solution of ethylene glycol was preferred as the noninflammable base stock after giving consideration to the inflammability results and the following factors (references 9 and 10):

- (1) Attack on packings by the solution

- (2) Volatility, flash point, and incendiary fire hazard of organic substance alone
- (3) Difficulty of formulating fluid of desired viscosity and viscosity index if base stock has too high a viscosity
- (4) Availability, on a large scale, of organic compound at time of investigation

The freezing points of both water and ethylene glycol are depressed when the liquids are mixed. The eutectic mixture freezes at approximately -65° F and contains 67 percent by volume of the glycol and 33 percent by volume of water. In order to offset water evaporation and the resulting rapid rise of freezing point and viscosity, a solution of 55 percent by volume of ethylene glycol and 45 percent by volume of distilled water was used. Furthermore, for adequate fire resistance, at least 40 percent by volume of water was found necessary. The freezing point of the selected base stock was -55° F.

The formulation that showed the most promise in the early investigations was called Hydrolube U and contained, in addition to the base stock, a viscosity-index improver of the polyalkylene-oxide-copolymer class, liquid- and vapor-phase corrosion inhibitors, and a copper deactivator. Selection of suitable inhibitors was made after examination of a large number of compounds and combinations (reference 10).

A second formulation was devised that showed less promise because of marked shear breakdown of the polymer during pump tests, greater corrosive effects, and evidence of hard tacky residue from the evaporation test. The main difference in constitution between this formulation, known as Hydrolube A, and Hydrolube U was the polymer used as the viscosity-index improver. A sodium polymethacrylate was employed in Hydrolube A. The composition and some of the properties of the two formulations, as well as those of Hydrolube U-4, are given in table III.

Inflammability Tests

The inflammability characteristics of the main components of Hydrolube U are indicated in table II. In addition, the Civil Aeronautics Administration (reference 15) conducted an investigation of such characteristics for several hydraulic fluids under conditions of simulated fire in flight and after crash. The fluids examined were released under pressures of 1000 and 3000 pounds per square inch and

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subjected to ignition by exhaust flames, hot exhaust stack, burning gasoline, and ignition spark. Fire after crash was simulated by the discharge of the fluids at a pressure of 3000 pounds per square inch into an electric arc and an oxyacetylene flame. By these tests, Hydrolube U was rated noninflammable. After expulsion of water from this fluid, however, a low flash point of 295° F was obtained (reference 15).

Chemical and Physical Tests

The behavior of water-base fluids in small-scale chemical and physical laboratory tests is reported by several laboratories (references 9, 10, and 16). Although these tests were not run under the same conditions, certain general conclusions are possible.

The viscosity of the Hydrolubes as formulated was designed to be equivalent at 130° F to that of AN-VV-O-366b fluid (a specification preceding AN-O-366), that is, 10 centistokes. At -40° F, however, the viscosity was nearly four times as great as the 500 centistokes this specification requires. The values easily met the SAE AMS 3150A requirement. Difficulties were encountered because of this rapid change of viscosity with temperature; a decrease in mobility of brakes and elevator boosters was noted on simulated systems at low temperatures (unpublished data compiled by the Aircraft Industries Association of America, Inc.).

Other low-temperature characteristics that are likely to limit performance are the freezing point of -50° to -60° F and the solubility of the various additives (reference 16 and unpublished data). The addition of another liquid component to lower the freezing point was suggested (reference 9); this addition might adversely affect the attack on packings and seals.

The effect of Hydrolubes on rubber packings has been studied in a number of laboratories (references 10, 16, and unpublished data). Some variation in results is reported; in general, however, it is believed that unless water is lost or operating temperatures are appreciably over 160° F, the action of Hydrolubes on packings is comparable to that of petroleum-base fluids.

The corrosive effect of Hydrolubes on metals encountered in the hydraulic system has been investigated (references 9, 10, and 16). Test procedure varied because no requirement is stated in specification 51F22 (reference 5). Indications were, however, that magnesium was heavily attacked, cadmium plate was slightly corroded, and copper was tarnished.

The foaming characteristics of Hydrolube U and A have not presented a problem. Hydrolube A showed less tendency to foam than AN-VV-O-366b fluid (reference 10) and Hydrolube U-4 caused no appreciable difficulty due to this characteristic (unpublished data).

A property, not part of the foaming characteristics, is the tendency for Hydrolube U-4 to hold air in suspension (unpublished data). This characteristic causes more difficulty in bleeding the hydraulic system than occurs with AN-VV-O-366b fluid.

The hard tacky residue from evaporation of Hydrolube A has already been mentioned. Even Hydrolube U and U-4 are thought to leave a less desirable type of residue (reference 16 and unpublished data) than most petroleum-base fluids. The residue from Hydrolube U-4 has the consistency of petrolatum, the effect of which is unknown (unpublished data). Better low-temperature properties, less attack on magnesium, and improvement of wear characteristics are among the objectives sought in the continued development of water-base fluids.

Pump-System Tests

Hydrolubes U and A were pump-tested by the Naval Research Laboratory using a Pesco 1P-349-N aviation gear pump in one hydraulic system and a Vickers PF-2713-10 aviation piston pump in another (reference 9). Both systems were otherwise similar and utilized standard aircraft parts. The tests were run at pressures of 1000 and 1500 pounds per square inch, at engine speeds of 1800 and 3600 rpm, and at temperatures of 100°, 140°, and 180° F. The pumps were periodically stopped to permit disassembly and inspection of the system. During the tests, samples of fluid were withdrawn from the reservoir in order to determine changes in viscosity, pH, color, development of precipitates, and other properties.

In the system equipped with the Pesco gear pump, the water-base fluids produced greater wear of the gears than would occur with the petroleum-base fluids. With higher temperatures, pressures, and speed conditions, the wear increased but no pump failures occurred.

With the Vickers pump, insufficient lubrication was likewise shown. Breakdown occurred because of ball-bearing failure either on the drive shaft or in the small bearings supporting the cylinder block. Better performance was obtained by circulating additional fluid through the ball bearings and by the addition of an antiwear additive, diamylammonium laurate, which is now a constituent of Hydrolube U-4.

Inferior wear characteristics for Hydrolubes U and U-4 have also been reported by other laboratories (reference 16 and unpublished data). In an experiment on Hydrolube U-4 presented in an unpublished report, a Pesco gear pump at a pressure of 3000 pounds per square inch and a temperature of 125° F was severely worn and decreased considerably in volumetric efficiency after 200 hours.

Failure of a Vickers pump ball-bearing assembly occurred in other tests on Hydrolube U-4 (unpublished data). In some of these cases, the system was operated for longer periods, for example, 296 and 308 hours. During the run operated for 296 hours, considerable fluid was lost by evaporation that resulted in a 23-percent increase in kinematic viscosity at -30° F after 250 hours of operation. The residual fluid showed no appreciable change in fire resistance.

The Naval Research Laboratory, having obtained somewhat encouraging results from bench pump tests on Hydrolubes A and U, continued the test program to the next stage, a hydraulic mock-up system including a number of hydraulically operated aircraft units. The tests, conducted at the Naval Research Laboratory and the Naval Air Material Center, again offered sufficient promise so that flight tests were made involving the two fluids.

Hydrolube A was tested in a Navy fighter-class plane for a period of 4 months. During this interval, approximately 100 hours of flight time accrued and the various units of the hydraulic system were frequently operated. At the conclusion of the test, the system still operated satisfactorily although on disassembly some threaded connections were found unduly tight.

Hydrolube U, having shown good results on most laboratory tests and low shear breakdown on the pump test, was deemed worthy of more extensive flight testing. Accordingly, this fluid has been tested for a year in 10 Navy fighter-class planes with low-pressure systems of 1500 pounds per square inch. Unpublished information obtained from the Bureau of Aeronautics indicated that planes with 300 flight hours have shown few leakage failures, although slight corrosion of magnesium parts occurred. Furthermore, Hydrolube U in selected planes with high-pressure systems of 3000 pounds per square inch gave satisfactory operation for at least 450 hours. The Vickers pumps, however, had deteriorated mechanically although still in operation (references 17 and 18).

According to unpublished information obtained from the Navy, Hydrolube U-4 was being employed in certain fighter planes as they came off the production line. In addition, three experimental models

were equipped with Hydrolube U-4 and operation thus far has been satisfactory. One change in the usual system was made, that of replacing the magnesium brake cylinders with aluminum brake cylinders. Navy experience indicates that the life of a Vickers pump, using Hydrolube U-4, is approximately 500 hours. Also, the life of high-pressure hydraulic systems in which Hydrolube U-4 is employed is about 80 to 90 percent of that when the petroleum-base fluid is used.

The Civil Aeronautics Administration has made flight tests with Hydrolube U-4 in C-47 and C-54 airplanes (unpublished data). The C-47 airplane has a low-pressure hydraulic system (less than 1000 lb/sq in.) and is reported to have operated satisfactorily for approximately 200 hours over a period of 6 months. Similarly, the C-54 airplane was operated for 200 hours during a period of 3 months. Hydraulic maintenance difficulties, not considered attributable to Hydrolube U-4, were encountered. Inspections of the magnesium brakes were made after 100 and 200 hours of operation. Nothing unusual in appearance was noted at the first inspection but after 200 hours, small white bubbles were noted near the surface of the magnesium. No evidence of pitting was observed. Samples of fluid were withdrawn after 66 and 114 hours of operation; no change in water content was found on analysis. A recent article (reference 19) indicates that the test on the C-54 airplane was stopped because of corrosion of magnesium equipment in the system. The article states that the test on the C-47 airplane was, at that time, still being conducted although it showed a similar tendency for attack on magnesium parts.

Advantages and Disadvantages

Hydraulic-fluid formulations based on ethylene glycol and water, such as Hydrolube U-4, show greatly decreased inflammability providing water is not lost. Performance, noted by various operators, suggests necessity for improvement of this type of fluid. At present, there is an indicated need for discovering means to decrease the wear in pumps, to lessen the attack on metals in the hydraulic system (especially on those more chemically active, such as magnesium), as well as to improve the low-temperature properties of the fluid.

SILICONE FLUIDS

Inflammability Tests

The liquid silicones of suitable viscosity and purity have received considerable attention as less-inflammable hydraulic fluids (references 7 to 9 and 20). In addition to high flash points and autogenous-ignition temperatures, some liquid silicones required from 55 to 85 percent of oxygen on the spray flammability test and appeared practically noninflammable in an incendiary-gunfire test. In general, the silicone polymers, particularly the polymethyl and poly(methyl, phenyl) siloxanes, were found to be less inflammable than petroleum-base fluids but slightly more inflammable than water-base fluids or halogenated compounds (references 15 and 21).

Chemical and Physical Tests

Extensive preliminary investigations of those physical and chemical properties pertinent to hydraulic fluids have been made by the Naval Research Laboratory (references 20 and 21) for a number of samples of silicone polymers submitted by various manufacturers.

One of the most desirable properties of the liquid silicones is the relatively small change of viscosity with temperature. As a result, a lesser value for A.S.T.M. slope is obtained for silicones than for usual hydraulic fluids.

The behavior of silicones when stored in metal or glass containers at ordinary or low temperatures is important because an increase in viscosity or formation of precipitates would decrease flow. Storage in tin orterne-plated cans or glass at 77° F in a dark room had little effect on the viscosity of most samples; less than a 5-percent increase was observed (reference 21). Furthermore, only insignificant differences in effect were noted between samples stored in cans and those in glass. Storage at temperatures of -40°, -50°, -60°, -70°, and -80° F for 24-hour periods indicated formation of crystals in most samples even at -40° F, although a number had pour points in the range of -50° to -80° F.

A suitable temperature for an accelerated oxidation test was found to be 392° F (reference 21); a higher temperature of 482° F caused rapid gelation of most samples. Silicones appeared relatively stable towards oxidation at temperatures to 302° F. The course of the oxidation was followed by observing change in viscosity and by

measuring the amounts of volatile acids and aldehyde produced. Both formic acid and formaldehyde were identified among these products. Some oxidation tests were run in the presence of metals likely to be present in systems lubricated. The metals used were copper, lead, aluminum, magnesium, and tin; the alloys used were bronze, cold-rolled steel, stainless steel, monel, babbitt, and Dowmetal H. Tin alone accelerated the oxidation, others inhibited the reaction; babbitt was the most effective.

The corrosive effect of silicone fluids on copper, lead, silver, magnesium, tin, babbitt, bronze, Dowmetal H, duralumin 17S-T, and cold-rolled steel was determined (reference 21) by a modification of the Federal Specification Board Method 530-31. The metal strips, after careful cleaning and polishing, were placed with the fluid in a sealed tube. The tube and the contents were heated in a thermostat-controlled bath at 212° F for a period of 24 hours. Other tests were made at 150° F for 168 hours (sealed tube), at 302° F for 213 hours, and at 392° F for 250 hours (oxidation tests), with extensive aeration in the last two instances. Results of the tests indicated that at 212° F, little attack on the metals was to be expected; at 302° F, only copper and its alloys would tarnish; at 392° F, copper, tin, lead, and their alloys were affected. Corrosion of the tin and the lead was the most serious.

Because water is a known contaminator in hydraulic systems, inhibition of the attack on metals by water-containing silicone fluids was desired. By application of previous experience with petroleum lubricants at least 10 suitable silicone-soluble corrosion and rusting inhibitors were obtained (reference 21). These inhibitors had to be soluble to at least -40° F and be effective at percentages of less than 1. The preferred group functioned at less than 0.5 percent.

Surface tensions and specific gravities of the several samples of silicone fluids were measured because of the intimate relation between these properties and the tendency of the fluids to creep over metal surfaces, to foam, and to emulsify with water (reference 21). The values determined for the surface tension were approximately 20 dynes per centimeter with two exceptions. These values were low when compared with those for typical petroleum oils, which were 28 to 32 dynes per centimeter. The specific gravities of the silicone fluids were mainly in the range of 0.95 to 0.99.

According to reference 21, the solubility of water in silicone fluids is in the order of 0.1 percent. Because a greater percentage of water might be expected in a hydraulic system, the question of the formation and the degree of stability of silicone emulsions arose.

The lubricity of an emulsion is known to be less than a homogeneous fluid because the lubricating film is discontinuous. Emulsions were prepared from submitted samples and from a sample subjected to oxidation at 392° F. Their stability was determined by noting the percentage of water separation after definite intervals of time. The effect of heat on the stability of certain emulsions was determined by observation of their behavior when prepared at 34°, 77°, 140°, and 180° F. Emulsions formed readily but in most instances were considered rather unstable and heat tended to render them less stable. The oxidized fluid formed a more stable emulsion.

Representative silicone fluids were investigated and were found to have less tendency to foam than standard petroleum-base hydraulic fluids. The need for an antifoam agent would therefore be unlikely if silicone fluids were to be used as hydraulic fluids.

Pump-System Tests

Because some of the chemical, physical, and inflammability investigations (reference 8) had indicated that several samples of silicone fluids contained volatile material of an inflammable nature, special samples, stripped of low-boiling-point constituents and with viscosities of 25 to 30 centistokes at 210° F, were obtained for study of their performance characteristics as hydraulic fluids (reference 20). The fluids were investigated in systems in which the Pesco 1P-349-N gear pump and the Vickers model PF-2713-10 constant-delivery aircraft piston pump were used. These small-size high-pressure pumps were considered a very useful means of determining the lubrication characteristics of the silicone fluids because nearly every type of wear would be represented, particularly in the Vickers pump.

The Pesco gear pump was equipped with nitrided gears running in high-lead bronze bushings. In table IV are presented the results of several runs conducted at the Naval Research Laboratory on a dimethyl-silicone-polymer fluid and an AN-VV-O-366b fluid using the system equipped with the gear pump. These results show that

(1) The weight losses of the gears and the bronze bushings were less with the silicone fluid than with the petroleum-base oil. A deposit of silicone resin on the bushings was believed to be responsible for the low wear, the resin acting as a protective coating.

(2) The change in viscosity using the silicone polymers was much less than when using the AN-VV-O-366b fluid. Little shear breakdown occurred with the silicones.

(3) Inspections of the system indicated that some gel had collected on the filter. In run P-25, the presence of gel was very pronounced. Serious clogging of the filter occurred after 500 hours of operation at 140° F and a pressure of 1500 pounds per square inch; the Purolator- or the Skinner-type filter without a bag was used. By simple visual examination of a fluid that was run for 100 hours, the gel was difficult to detect but could be seen by microscopic inspection.

When the Pesco gear pump was fitted with cast-iron bushings, repeated failures occurred even at a pressure as low as 600 pounds per square inch at a temperature of 180° F. Considerable gel had formed and the fluid was black because of suspended finely divided iron. The cast-iron bushings were badly worn.

This contrast in wear characteristics of steel-to-bronze and steel-to-cast-iron, when lubricated by silicone fluids, led to an extensive study of the behavior of these fluids with 156 combinations of metals and alloys by means of a slider and a plate (reference 20). Comparison was also made of the lubrication characteristics of a petroleum-base oil N.S. 1047 with these metal combinations. At least 81 satisfactory bearing-metal combinations were found that could be lubricated by means of the silicone fluid.

On the tests with the Vickers piston pump, failure due to the inadequate lubrication of the steel knuckles in the universal joint of the drive shaft occurred on two runs. At a pressure of 1500 pounds per square inch and at temperatures of 140° and 100° F, failure occurred at 65 and 80 hours, respectively. The fluid contained suspended particles of iron. When bronze knuckles were substituted for the steel type, however, the pump performed satisfactorily for 650 hours at a pressure of 1500 pounds per square inch and a temperature of 180° F.

Studies on the behavior of dimethyl-silicone-polymer fluids (references 22 and 23) and poly(methyl, phenyl) silicone polymer (reference 23) in the lubrication of unilaterally loaded journal bearings have been made. The results show that lubrication of common bearing metals, other than steel-to-steel or steel-to-cast-iron, may be successfully accomplished with these fluids. The protective silicone resin coating, formed by a slow break-in or stabilization of the bearing in the fluid for a long time at 300° F or above, materially aids in the lubrication process.

Two difficulties due to characteristics of liquid silicones were encountered in the pump systems (references 9 and 20): the tendency of the fluid to leak at joints in the connections and the tendency to extract plasticizer from the packings, which caused the O-rings to shrink, become brittle, and permit leakage. These difficulties were overcome by using the silicone fluid under test as a plasticizer in O-rings made of synthetic rubber containing graphite. A special cement was obtained from commercial sources and was used as a pipe-thread sealing compound, thus making the use of these fluids feasible without undue leakage.

Another satisfactory commercial packing for silicones has been developed, as noted in unpublished data from the General Electric Company.

Advantages and Disadvantages

Polymethyl and poly(methyl, phenyl) siloxanes of suitable viscosity for use as less-inflammable hydraulic fluids show less change of viscosity with change in temperature than do other liquids such as the petroleum-base fluids. The silicones are distinctly less inflammable, are not readily oxidized, and are not particularly corrosive. Several available inhibitors of corrosion have been found. Little tendency to form permanent emulsions with water has been exhibited by silicones unless they are oxidized.

Disadvantages shown in aircraft hydraulic systems have resulted from a lack of lubricity of these silicones for some metal combinations, notably steel-to-steel or steel-to-cast-iron although many other pairs were found satisfactory. The silicones tended to leak at the pipe joints because of low surface tension of the fluid and to extract the plasticizer from O-rings. Means of overcoming these difficulties have been reported by the Naval Research Laboratory (references 9 and 20).

Halogenated esters such as phosphates, dicarboxylates, or carbonates added to silicones might form suitable solutions or even copolymers, thus decreasing the inflammability. Certain halogenated hydrocarbons when mixed with silicones might form a useful less-inflammable fluid.

ESTER-BASE FLUIDS

Several ester types have been investigated as possible important base constituents for less-inflammable hydraulic fluids. Of chief

interest were those members of each type that had sufficiently high boiling points to be less volatile than the hydrocarbon or other components of the petroleum-base hydraulic fluid meeting the AN-VV-0-366b specification. Ester types investigated were phosphates, borates, carbonates, and dicarboxylates (references 7, 11, 14, and 24 to 27).

The open-cup fire point of the several classes of esters investigated is shown in the following table of compounds, each having a viscosity of 4.0 centistokes at 100° F. The data presented are taken from reference 16 from plots of viscosity as a function of boiling point and as a function of fire point.

Ester type	Boiling point at 760 mm (°F)	Open-cup fire point (°F)
Naphthenic hydrocarbons	536	290
Alkyl carbonates	599	318
Alkyl borates	614	290 (decomposed)
Alkyl dicarboxylates	622	330
Alkyl phosphates	636	420

Thus, the alkyl phosphates apparently offer the greatest promise of the four ester types in respect to their degree of inflammability as determined by the open-cup fire-point method. Furthermore, reference 16 indicates that phosphates of the several classes of esters have the highest fire point for a given boiling point.

Phosphate Esters

A number of phosphate esters were investigated by the Air Materiel Command (references 16 and 25) and included the following types:

Tri- <u>n</u> -butyl phosphate	Tri- <u>n</u> -octyl phosphate
Tri- <u>n</u> -amyl phosphate	Tri-2-ethylhexyl phosphate
Tri-2-ethylbutyl phosphate	Trimethoxyethyl phosphate
Tri- <u>n</u> -hexyl phosphate	Tributoxyethyl phosphate

Data presented in reference 16 concerning the flash points of these esters indicate most of the values to be twice as high as the 200° F minimum required of a petroleum-base fluid. Tri-2-ethylhexyl phosphate and tributoxyethyl phosphate, however, possess the disadvantage of viscosities greater than 500 centistokes at -40° F (reference 16).

The Naval Research Laboratory (reference 7) investigated the characteristics of a group of phosphate esters, subjecting them to the following inflammability tests: flash and fire points, autogenous-ignition temperature, and oxygen demand. The phosphorus esters investigated by this research group included:

Trimethyl phosphate	Diphenyl mono(p-tert-butylphenyl) phosphate
Tributyl phosphate	Di-(o-chlorophenyl) monophenyl phosphate
Trioctyl phosphate	Tetrabutyl pyrophosphate
Tricresyl phosphate	Hexamethyl tetraphosphate
	Diethylphenyl phosphonate

Pure trimethyl and tricresyl phosphate and hexamethyl tetraphosphate showed somewhat favorable inflammability characteristics.

Chemical and physical tests. - Considerable testing at the request of the Air Materiel Command, particularly of the tri-n-butyl and tri-n-hexyl esters individually or as components of less-inflammable hydraulic fluids, has been completed (references 16 and 25). In some cases, di-(2-ethylhexyl) sebacate was included in blends as an antitack component. Important results from compositions in which these substances were employed will therefore receive greatest attention in the subsequent discussion.

The solubility of water in the esters or vice versa presented difficulty particularly in the case of phosphates of low molecular weight. Trimethyl, triethyl, and trimethoxyethyl phosphates are miscible or very soluble in water at room temperature. Prolonged exposure to water may even lead to considerable hydrolysis of esters of low molecular weight. From experimental determinations, water was found to dissolve in a number of phosphate esters at $75 \pm 5^\circ \text{F}$. (reference 16) as follows:

Phosphate	Percent by weight of water dissolved
Tri-n-butyl	6.5
Tri-n-amyl	4.0
Tri-n-hexyl	2.8
Tri-n-octyl	1.2
Trimethoxyethyl	Soluble
Tributoxyethyl	6.9

In order to prevent the formation of ice at -40°F when tri-n-butyl phosphate was to be a component of the fluid, it was found advisable to include another member of this class, for example, 30 percent by weight of tri-n-hexyl phosphate or a dibasic-ester

type, such as 20 percent by weight of di-(2-ethylhexyl) sebacate. A calculated amount of Acryloid 855 (ester polymer commonly used as a viscosity-index improver) was then added. These components were miscible with each other at ordinary and low temperatures.

Another property studied for sample fluids was tackiness. Results were observed for tests conducted according to the procedures of specifications AN-VV-O-366b or O.S. 2943 (superseded by 51-F-21). This characteristic was attained in the desired amount when tri-n-hexyl phosphate or di-(2-ethylhexyl) sebacate esters were incorporated in the fluids. Tri-n-butyl phosphate in 12 percent by weight of Acryloid 855 did not pass the AN-VV-O-366b requirements for this property.

Fluids formulated with the phosphate esters have shown considerable oxidation stability when subjected to procedures of the AN-VV-O-366b or O.S. 2943 specifications. Additional stability has been noted for one fluid after the addition of an amine type of oxidation inhibitor (references 16 and 25). Even 0.2 percent by weight of Methyl H1 (an oxidation-corrosion inhibitor) in the blend designated PRL 2476 (a hydraulic fluid composed of 12.2 percent by weight of Acryloid 855, 17.6 percent by weight of di-(2-ethylhexyl) sebacate, and 70.2 percent by weight of tri-n-butyl phosphate) caused some improvement in neutralization number and also in the amount of copper lost when this blend was subjected to the O.S. 2943 oxidation and corrosion test.

Hydraulic fluids formulated with high amounts of phosphate esters tend to swell the rubber packings used in the hydraulic system. Results reported in reference 16 indicate that the alkyl types of high molecular weight, such as tri-n-hexyl, tri-2-ethylhexyl, and tri-n-octyl phosphates, caused less swelling of a butadiene-acrylonitrile rubber than did the lower-molecular-weight tri-n-butyl phosphate. Sulfide rubbers are much less affected by swelling than the butadiene-acrylonitrile rubber. Research is being conducted by industrial groups to find suitable materials for packings and gaskets.

Pump-system tests. - Blend PRL 2476 (reference 25) was tested primarily for wear by means of a hydraulic system that included a Pesco 349 gear pump and a Vickers Model C-167-G relief valve. Runs 162 and 165 in table V were made according to the procedure in the O.S. 2943 specification, which required a pump speed of 3600 rpm.

Inspection of the results, after considering that no oxidation inhibitor was present in PRL 2476, leads to the following observations:

(1) The wear shown, utilizing the O.S. 2943 or O.S. 1113 (superseded by 51-F-23) procedure, was greater than that for a hydrocarbon-base fluid. The steel gears appeared to undergo the greatest loss. In run 163, conditions of pressure and temperature were made more severe than required by the specifications and resulted in excessive wear in the steel gears and normal wear for the bronze type.

(2) Neutralization numbers were higher than normally encountered with hydrocarbon-base fluids, a result attributed to the absence of any oxidation inhibitor. Results of run 163 would indicate that at higher temperature and pressure greater oxidation took place.

Borate Esters

This class of compounds has been considered as a constituent of less-inflammable hydraulic fluids (references 16 and 25). Two alkyl types, tricapryl and tri-2-ethylhexyl borates, were prepared from trimethyl borate by an alcoholysis reaction and their properties significant to the problem were investigated (reference 25).

Present research results indicate that pertinent organic borates decompose as their temperature is being raised towards the boiling point unless reduced pressure is maintained. This behavior is believed to account for the fact that the fire point for alkyl borates, given in the first table of this section, was lower than might otherwise be expected.

Ease of hydrolysis of the borates may present a problem. The two alkyl borates previously mentioned were at least 90-percent hydrolyzed without a catalyst within 45 minutes. The addition of 15 percent by weight of Acryloid 855 to these borates permitted only slightly less hydrolysis of these esters. Borates prepared by using neopentyl alcohol or 2,4-dimethyl-3-pentanol, in which branching occurs near the ester linkage, hydrolyzed quite slowly, if at all; unfortunately, the melting points of these two esters were found to be quite high (reference 25).

Until organic borates stable to heat and moisture and with suitable physical properties can be produced, this type of ester can be given only secondary consideration.

Carbonate Esters

Research on several carbonic acid esters has been reported. The Naval Research Laboratory (reference 7) has expressed an interest in diethylene glycol bis(n-butyl carbonate) and diethylene glycol bis(2-n-butoxyethyl carbonate). These two esters had an exceptionally high oxygen demand of 85 percent although results of flash, fire, and autogenous-ignition-temperature tests disagreed with this value.

Other investigators (reference 16) have also shown considerable interest in this type of compound as a base stock for a less-inflammable hydraulic fluid. Properties of carbonate esters indicate different degrees of usefulness of the samples examined for application in this particular field. A concise account of the results reported in reference 16 will be presented.

Esters of this class were obtained or made by utilizing the following alcohols:

Alcohols in dialkyl and dicycloalkyl types

Ethanol
n-Butanol
n-Pentanol
n-Hexanol
n-Octanol
Cyclopentanol

Alcohols in cyclic carbonates

1,2-Propanediol
2,3-Butanediol
2,3-Pentanediol
2-Methyl-2,4-pentanediol

Mixed alcohols in dicarbonates

n-Butanol, diethylene glycol
n-Butoxyethanol, diethylene glycol
Phenol, diethylene glycol

Several distinctive properties of these compounds and experimental fluids should be emphasized. Carbonate esters with a kinematic viscosity of 4 centistokes at 100° F were shown to have higher boiling points and higher fire points than petroleum fractions of the same viscosity (reference 16).

Another quality in which carbonates show superiority along with the trialkyl phosphate type is in the viscosity-temperature relation. Only the dialkyl and monocyclic carbonates of low molecular weight show less-satisfactory values of A.S.T.M. slope for the range of 210° to -40° F than does a petroleum fraction found in a petroleum-base hydraulic fluid.

An undesirable property found for most carbonate esters, with the exception of the dicarbonate type, was the extent of hydrolysis. For the dicarbonate type, the amount of hydrolysis observed was less than 0.5 percent by weight at the end of 48 hours. The range for the dialkyl and cyclic carbonates was 21 to 63 percent. This tendency of carbonate esters to hydrolyze while in service might change viscosity, volatility, freezing point, oxidation rate, and other characteristics of the fluid.

Another property of the carbonates investigated was the effect on butadiene-acrylonitrile rubbers A and C. Di-n-amyl and di-n-octyl carbonates, butoxyethyl diglycol carbonate, and n-butylene-2-carbonate caused greater rubber swelling than the 6 percent permitted by the AN-VV-O-366b specification; the di-n-octyl derivative showed the most promise (reference 16). The opinion has been expressed that the dialkyl-type carbonates of higher molecular weight might give the least rubber swelling although greater inflammability would result.

A suitable polymer that would improve the viscosity properties of possible blends containing carbonate esters is needed. At least two carbonate esters, the di-n-hexyl and the di-n-octyl carbonates, have viscosities that suggest their possible use in less-inflammable hydraulic fluids. Acryloid 858 was found to be only partly satisfactory as the polymer in blends that contained either of these esters. At -25° F, the fluid containing di-n-hexyl carbonate gave a cloudy solution and the one containing di-n-octyl carbonate froze.

Dicarboxylate Esters

The investigation of the suitability of this class of compound, particularly the aliphatic types, as components of less-inflammable hydraulic fluids is reported in references 7, 16, 24, 26, and 27. This particular type of ester has received some attention not only as a secondary but also as a principal constituent.

Inflammability tests. - At present, data on inflammability for numerous examples of this type of ester (references 24 and 26) indicate that a number of adipates, glutarates, azelates, and sebacates that are

esterified with branched-chain alcohols show higher values than O.S. 2943 hydraulic fluids for flash and fire points and autogenous-ignition temperatures. In contrast, the values found for these diesters when tested for inflammability by the oxygen-demand method offered only slight improvement over petroleum-base hydraulic fluids, such as AN-VV-O-366b or O.S. 2943 types (reference 7).

Fire-point data reported in reference 16 were similar to data of the Naval Research Laboratory (reference 26) for corresponding esters tested, for example, di-(2-ethylhexyl) adipate and di-(2-ethylhexyl) sebacate. Some improvement of the inflammability characteristics of a hydraulic fluid appears possible through the use of such dicarboxylic esters.

Chemical and physical tests. - Many of these diesters actually show a much better viscosity-temperature relation than do the petroleum fractions utilized in petroleum-base hydraulic fluids (references 24 and 26). This characteristic is insufficiently pronounced to eliminate the use of a suitable polymer in hydraulic-fluid formulations. Some dicarboxylic esters with viscosities of 3 to 13 centistokes at 100° F and an A.S.T.M. slope of 0.7 to 0.8 might prove suitable for use (reference 24).

The hydrolytic stability of the dicarboxylic acids esterified with branched-chain alcohols was determined (reference 26). Such esters appeared quite stable when a 10-gram sample was boiled with 50 milliliters of water for 24 hours.

The oxidation characteristics of a number of dicarboxylic acids esterified with branched-chain alcohols were determined (references 24 and 27). In both investigations, inhibitors were found necessary to reduce the oxidation to acceptable amounts.

The extent of rubber swelling caused by the dicarboxylic acid esters is reported in reference 24. The butadiene-acrylonitrile rubber C was employed with diesters having molecular weights of 230 to 426. Higher swelling values for this rubber were observed with these dicarboxylic acid esters than were noted with the AN-VV-O-366b specification fluid.

Attention should be drawn to the fact that two investigators (references 24 and 27) have suggested this type of ester as a possible main constituent of a less-inflammable fluid. The examples mentioned in reference 27 are di-(3-methylbutyl) adipate and azelate; the example mentioned in reference 24 is di-(2-ethylhexyl) sebacate.

Advantages and Disadvantages

Of the four types of ester discussed, the phosphates seem to possess a lesser degree of inflammability than do the dicarboxylates, borates, and ordinary carbonates investigated thus far.

Results of considerable research indicate that formulations having adequate viscosity properties, particularly at low temperatures, are more readily obtained from phosphate esters (for example, tri-n-butyl, tri-n-hexyl, tri-2-ethylhexyl, and tri-n-octyl phosphates) than from the other types of ester.

The usual phosphate esters do not appear to be as subject to hydrolysis as the other ester types, especially borates and carbonates. Instability at elevated temperatures has been shown by the borates even without any added moisture being present.

Pump-system tests with a Pesco 349 gear pump on a phosphate blend showed wear and the need for an oxidation inhibitor for this type of less-inflammable fluid. Ester fluids must be properly inhibited against corrosion as well as oxidation. Attack on rubber packings by phosphates, carbonates, and dicarboxylates is greater than for a standard petroleum-base hydraulic fluid.

As a possible means of decreasing the inflammability of the several classes of esters, the formation of halogenated derivatives is reportedly in process and may be ultimately successful. The use of a less-inflammable diluent with suitably halogenated esters and their derivatives represents still another possibility. Finally, a less-inflammable viscosity-index improver might be found that would aid in the achievement of decreased inflammability for the ester-type hydraulic fluids.

HALOGENATED-BASE FLUIDS

The compounds chiefly considered are those expected to perform a snuffer function. A snuffer constituent is a noninflammable substance with a somewhat higher vapor pressure than the other components of the hydraulic fluid; when the fluid is exposed to an ignition source, some of the snuffer is vaporized and then blankets the remainder, thus excluding air and preventing combustion (reference 28). Actually, the water of the water-base fluids probably functions in part as a snuffer constituent.

Chloro, fluoro, and chlorofluoro organic substances have received the greatest attention as halogenated snuffer constituents (references 28 and 29). Few such compounds known to possess the required properties for use as less-inflammable hydraulic-fluid components are as yet available on a commercial basis. As a result, a considerable amount of research is being done to find the most favorable classes and structures among halogenated compounds for this purpose. This research may eventually lead to discovery of suitable components for an acceptable noninflammable fluid.

The interest of various laboratories in completely halogenated compounds as a means of attaining noninflammable rather than less-inflammable hydraulic fluids is of special note. A recent report (reference 11) offers suggestions in regard to theoretical properties and practical possibilities in this field. An interesting discussion of pertinent properties of some fluorinated compounds is presented. In order to improve viscosity characteristics of a fully fluorinated compound, the suggestion is made that each molecule have trifluoromethyl side chains. Compounds suggested for consideration are polymers of perfluoropropylene or perfluorobutylene, substances such as perfluorobutylene ether and thioether, and perfluorinated dibutyl-methylamine and dilaurylmethylamine.

According to unpublished information and references 11, 27, and 29, members of the following general types of halogenated substance have been or are being considered for their suitability as snuffer components:

Perchloro types

- Straight-chain saturated aliphatics
- Straight-chain olefins
- Straight-chain diolefins

Perfluoro types

- Straight-chain saturated aliphatics
- Polymers of olefins
- Alkyl cyclohexanes
- Alkyl naphthalanes
- Fused-ring aromatics
- Fused-ring alicyclic compounds
- Kerosene
- SAE 10 oil

Chlorofluoro types

Straight-chain saturated aliphatics
Straight-chain olefins
Polymers of straight-chain olefins
Chloro-(perfluoroalkyl) benzenes
Chlorofluoroalkyl benzenes
Chloro-(chlorofluoroalkyl) benzenes
Chlorofluoro-(perfluoroalkyl) benzenes
Chlorofluoroalkyl phenyl ether
Chlorofluorocyclohexenes
Chlorofluorocyclohexyls, di and tricyclohexyls

Partly fluorinated types

Mono-(fluoroalkyl) glycol ether
Di-(fluoroalkyl) glycol ether
(Perfluoroalkyl) benzenes
(Perfluoroalkyl) phenyl alkanol
Alkyl-(perfluoroalkyl) phenyl ether
Perfluoroalkyl-(perfluoroalkyl) phenyl ether
(Perfluoroalkyl) benzoate
Diester of perfluoroaliphatic dicarboxylic acids

Miscellaneous types

Dicarboxy derivative of polychlorinated bicyclohydrocarbons
Partly fluorinated alkyl or alkylaryl phosphate
Tetra (fluoroalkyl) silicate
Fluorinated alkyl silicones
Chlorofluoro triazine
Bromo-(perfluoro) benzene
Bromochloro-(perfluoro) benzene

Some of these members of the numerous types of halogenated organic chemical, chiefly the polymers and the perfluoro compounds, may possibly find application as constituents of a noninflammable hydraulic fluid.

Requirements of Halogenated Snuffers

A satisfactory snuffer should possess the following properties:

- (1) Adequate solubility in mineral oil or other selected base stock over range of temperature to which it may be exposed

- (2) Boiling point high enough (above approximately 400° F) to prevent serious evaporation losses but low enough to allow snuffer action
- (3) Resistance to hydrolysis and oxidation and the resulting formation of acidic materials that may corrode metals
- (4) Ability to lubricate and not cause undue wear of pumps
- (5) Should not cause excessive swelling or shrinkage of hydraulic-system packings
- (6) Snuffer and its thermal-decomposition products should not be too toxic

Based on results of investigations thus far, a number of chloro and chlorofluoro compounds have shown more promise in the fulfillment of most of these requirements than the known types of fluorinated substance, particularly the fluorocarbons. For example, the fluorocarbons generally show inadequate solubility in the hydrocarbons found in mineral-oil fractions that are used in petroleum-base hydraulic fluids; research of a fundamental nature on solubilities of fluorocarbons is being conducted and has been reported in references 30 and 31. Furthermore, fluorocarbons exhibit great changes in viscosity with change in temperature (references 29 and 32), a difficulty that possibly may be overcome by selection of compounds of a suitable type. At present, the cost of commercially available chlorofluoro and fluoro compounds is very high.

Formulations of Snuffer-Containing Fluids

Less-inflammable hydraulic fluids formulated with snuffer constituents have been investigated by various organizations (references 28 and 29). A large number of the halogenated compounds considered in one of these investigations has been supplied through the Purdue Research Foundation (reference 33). The research authorized by the military services on the utilization of compounds as snuffers has been performed in the Petroleum Refining Laboratory at the Pennsylvania State College and in the experimental laboratories of the Air Materiel Command at Wright-Patterson Air Force Base, hereinafter denoted as PRL and AMC, respectively.

Preliminary testing. - The nature of the snuffer-containing fluids developed at PRL was governed by the inflammability limits set up by the SAE AMS 3150A specification and performance requirements of the AN-O-366 specification. The snuffers selected for the

fluids were: hexachlorobutadiene, chlorobis(trifluoromethyl) benzene, dichlorobis(trifluoromethyl) benzene, and Fluorolube Fore Shot. Only hexachlorobutadiene was comparatively reasonable in price and sufficiently available for thorough examination, which included a Pesco gear-pump test of one formulation that contained this material.

The one objectionable property of hexachlorobutadiene is its freezing point of -7°F (-22°C). Early inflammability tests indicated that a minimum of 50 percent by weight of this snuffer was required for desirable results. Formulations containing at least this amount of the chlorinated compound were investigated. Despite the high freezing point of this snuffer, some fluids exhibited reasonable performance on the low-temperature stability test. This behavior is explained as being due to a super-cooled-liquid effect of hexachlorobutadiene.

The selection of these compounds was based on boiling points, solubilities in probable constituents of the finished fluid, rubber-swelling characteristics, foaming tendencies, known toxicities, and wear properties as determined by means of the Shell Four-Ball Wear Tester (reference 29). Composition and properties of some of the formulations examined by PRL are given in table VI.

During the investigation conducted by AMC (reference 28), six less-inflammable snuffer-containing hydraulic fluids were formulated and tested. Each fluid contains Fluorolube Fore Shot (light ends of polymerized trifluorovinyl chloride) in an appreciable amount as well as an additional snuffer component that is as yet commercially unavailable; these formulations are at present quite expensive. The compositions are presented in table VII.

The development of these six formulations was preceded by an investigation of a number of halogenated compounds considered as possible components of less-inflammable hydraulic fluids. Table VIII shows the properties of a number of snuffer-type halogenated compounds. Additional compounds of this type that were considered (reference 28) are: (1-difluoro-2-difluoroethyl) ethylene glycol, di-(1-difluoro-2-difluoroethyl) ethylene glycol, Fluorolube (polymerized trifluorovinyl chloride), Fluorolube Fore Shot, trifluoromethyl benzene, perfluoro-m-dimethylcyclohexane, perfluoroheptane, fluorinated SAE 10 oil, and two polymers of trifluorochloroethylene. Only the Fluorolube Fore Shot, previously mentioned, was selected from the last group.

A number of tests to select the most suitable compounds to be used as snuffers in the less-inflammable hydraulic fluids were reported in addition to the information presented in table VIII. A brief résumé of the more important results of the preliminary examination follows:

(1) The extent of hydrolysis of possible snuffers did not seem very serious. The 3,3-difluorotetrachloropropene and the chloro-1-(trifluoromethyl)-4-(chlorotetrafluoroethyl)benzene appeared slightly unstable; in both cases, the active halogen may have been due to an impurity.

(2) Swelling of standard rubber packings F, H, and K by a number of the chlorofluoro compounds investigated so far was greater than that caused by either the high- or low-reference fluids. Fluorolube Fore Shot and perfluoromethylnaphthalene caused a smaller amount of swelling of the packings.

This rubber-swelling characteristic of halogenated snuffer compounds presents a problem that many investigators believe may require the use of special packings in the hydraulic system.

(3) Solubility of the fluorochloro types of organic compound in the Acryloid viscosity-index improvers, such as HF-855 and some common solvents including specification AN-O-366 hydraulic fluid, was much better than for the fluorocarbons.

(4) Flash points, fire points, and viscosities of favorable components and mixtures of these components were determined.

The results of these tests led to a selection of Fluorolube Fore Shot, tetrachlorotetrafluoropropane, chlorobis(trifluoromethyl)benzene, dichloro-(trifluoromethyl)benzene, dichlorobis(trifluoromethyl)benzene, 3,3-difluorotetrachloropropene, and chloro-1-(trifluoromethyl)-4-(chlorotetrafluoroethyl)benzene as snuffers for the six less-inflammable formulations.

Inflammability tests. - In the PRL investigation (reference 29), inflammability tests were limited to hexachlorobutadiene-containing snuffer-type fluids because of insufficient supplies of other snuffers investigated. The results of these inflammability tests on several formulations are presented in table IX. Both IO 2738 and IO 2739 appear to be essentially no more inflammable than HS-1 (the fluid designed to function as a standard for such tests) and much less inflammable than the petroleum-base fluid AN-VV-O-366b. None of the test samples, of course, showed the reduced inflammability of hexachlorobutadiene.

The compositions of the six less-inflammable formulations tested by AMC (reference 28) are presented in table VII. Each fluid contains approximately 51 percent by weight of the petroleum-base stock made up of 80 percent by volume of XCT White Oil and 20 percent by volume of Voltesso 36. Acryloid HF-855 is the viscosity-index improver and Paranox 441 is the oxidation-corrosion inhibitor. The last blend contained slightly more than half the amount of Fluorolube Fore Shot present in the others. The remainder of each blend is made up of one of the other snuffers selected.

Inflammability tests of these blends, in accordance with the SAE AMS 3150A specification (table X), indicated a high degree of fire resistance. The only evidence of any tendency to burn was observed in the high-temperature ignition test. Such results were comparable to those obtained by PRL (reference 29).

Chemical and physical tests. - The composition, the viscosity, and the pour points of a number of experimental less-inflammable fluids containing a common viscosity-index improver, the light hydrocarbon fractions of petroleum-base fluids and one or more of the four snuffers chosen for the investigation are given in table VI. Although formulations containing Fluorolube Fore Shot received considerable attention, other blends showed better characteristics. Blends PRL 2743, 2744, 2745, and 2746, of which Fluorolube Fore Shot was a constituent, exhibited unsatisfactory low-temperature properties that would exclude their selection by the United States Air Force. Disregarding the cost factor, even commercial air-transport companies might be reluctant to use the two better formulations containing Fluorolube Fore Shot, PRL 2744 and PRL 2745, because of their tendency to cloud near -40° F.

Sample PRL 2738, one of the four blends containing only hexachlorobutadiene as the snuffer, showed desirable characteristics. The formulations PRL 2737 and PRL 2810 that contain the same snuffer in larger amounts are distinctly less inflammable and would be more expensive.

Blend PRL 2739 actually employed a smaller percentage by weight of hexachlorobutadiene than the other blends of this group. The more viscous of the two white oils, Voltesso 36, was replaced entirely by 2-ethylhexyl sebacate, which, because of its high boiling point and oxygen content, exhibits a slight decrease in inflammability. This substitution caused an increased viscosity for blend PRL 2739 at low temperatures; the value found was 971 centistokes at -40° F.

Although this result is too high for requirements of the AN-O-366 specification, it is well within the limit for the SAE AMS 3150A type of less-inflammable hydraulic fluid, which is 2000 centistokes maximum at -40° F. Blends PRL 2738 and PRL 2739 were therefore thought suitable types for further evaluation (reference 29).

In the examination of less-inflammable hydraulic fluids (reference 29), PRL conducted investigations that sought to establish the most applicable unit for the viscosity of the fluid for hydraulic systems of airplanes. In accordance with several specifications, viscosities generally have been expressed in kinematic terminology. Results of the research by PRL have established the fact that the viscosity, particularly for a number of low-temperature applications of petroleum-base or halogenated hydraulic fluids in aircraft, should be expressed in absolute terms, that is, as centipoise units.

Oxidation and corrosion tests on the selected blends with suitable inhibitors were run in accordance with the procedure and the technique stated in specification AN-VV-O-366c, which was superseded by AN-O-366 (table XIV of reference 29). This method required the use of 100 milliliters of test fluid, which was heated for 168 hours at 250°±2° F. The catalyst combination consisting of 2 square inches each of copper, steel, aluminum, magnesium, and cadmium-plated steel was placed in the tube with the fluid. Air was passed through at 10±1 liters per hour and each large tube was fitted with a condenser to prevent loss of fluid.

The results of this test on blends PRL 2738 and PRL 2739 when known oxidation and corrosion inhibitors were employed are shown in table XI. Also included are data for hexachlorobutadiene, Fluorolube Fore Shot, and monochloroxylen hexafluoride (monochlorobis(trifluoromethyl)benzene). The AN-O-366 requirements for this test are also given in table XI (according to this specification, air is passed through the fluid at only 5 liters/hr).

Inspection of the data from the tests on these compounds and blends indicated:

(1) Oxidation of hexachlorobutadiene was negligible but there was considerable corrosion of magnesium and aluminum and slight corrosion of copper.

(2) Fluorolube Fore Shot appeared very stable to oxidation and caused little corrosion.

(3) Monochloroxylene hexafluoride indicated too high a volatility.

(4) Common commercial oxidation and corrosion inhibitors employed did not adequately regulate these properties for blends PRL 2738 and PRL 2739 (note table XI, columns 4 to 17). Copper and magnesium corrosion appeared particularly difficult to control and neutralization-number increase and color change seemed unusually hard to reduce to reasonable values.

(5) The most favorable combination of inhibitors for PRL 2738 against corrosion appeared to be Paranox 441, Paranox 512, and Dupont metal deactivator. Tetrabutyl tin added to this combination reduced both the neutralization number and color change, as shown by column 28. Further research on this phase of the program will be done.

Bench tests made on the six formulations (table VII) investigated by AMC (reference 28) indicated that the chief defect of these formulations was the amount of rubber swelling observed (table XII). This characteristic was not determined for four of the fluids.

Pump-system tests. - The hydraulic-fluid specifications described in reference 6 required pump and shear-stability performance of the less-inflammable hydraulic fluid to be comparable with or superior to an AN-VV-0-366 specification hydraulic fluid. As a result, tests were conducted by PRL (reference 29) on a snuffer-containing fluid with a Pesco 349 gear pump, loaded by means of a Vickers model 167G relief valve. The tests were conducted under mild and severe conditions according to the following requirements:

Pump-test conditions		
	Mild	Severe
Pressure, (lb/sq in.)	1000	2500
Speed, (rpm)	1800	3600
Temperature, (°F)	100	200
Time, (hr)	100	100

The tested fluid, PRL 2785, had the following composition:

Component	Percent by weight
Acryloid 855	5.3
XCT White Oil	4.2
Voltesso 36	43.1
Hexachlorobutadiene	46.8
Paranox 441	.4
Paranox 512	.07
Dupont metal deactivator	.05

The results of pump tests on this blend and specification AN-VV-O-366b types, with and without 1-percent tricresyl phosphate, are given in table XIII. Gear-wear values for PRL 2785 were less than for the specification AN-VV-O-366b fluid and slightly higher than those for a similar fluid containing the tricresyl phosphate.

According to reference 29, PRL 2785 would show difficulty in meeting the oxidation requirement of specification AN-O-366 in respect to change of neutralization number and color. A further difficulty might occur at low temperatures if the hexachlorobutadiene crystallized.

No pump tests were possible at this time on blends containing monochlorobis(trifluoromethyl)benzene and dichlorobis(trifluoromethyl)benzene. The quantities of these substances on hand or obtainable were insufficient for the production of the required blends. As a result, further research on snuffer-type fluids may be expected (reference 29).

Pump tests of the six less-inflammable fluids (table VII) have been delayed because of the limited quantities of snuffers available. Lubrication in connection with steel-to-steel and steel-to-bronze is a matter of considerable interest. Little difficulty due to shear breakdown of the fluids is expected with these tests because of the amount of petroleum-base oil and the nature of the viscosity-index improver present in each fluid.

Advantages and Disadvantages

The use of halogenated compounds, chloro or chlorofluoro in nature, as snuffer constituents of less-inflammable hydraulic-fluid formulations has been shown both possible and useful. Perfluoro compounds tested thus far have displayed inadequate solubility with other probable constituents of less-inflammable fluids. Perfluoro compounds are also being considered for use in noninflammable hydraulic fluids.

Halogenated compounds that have been shown useful thus far as snuffer constituents are: hexachlorobutadiene, chlorobis(trifluoromethyl)benzene, dichloro(trifluoromethyl)benzene, dichlorobis(trifluoromethyl)benzene, chloro-1-(trifluoromethyl)-4-(chlorotetrafluoroethyl)benzene, Fluorolube Fore Shot, tetrachlorotetrafluoropropane, and 3,3-difluorotetrachloropropene. At least 50 percent by weight of the snuffer component or components were deemed necessary in the fluids. Favorable results have been obtained from chemical and physical tests on formulations containing one or more of these snuffers.

Principal obstacles encountered by PRL in tests on formulations were the low-temperature properties of some test fluids and the control of oxidation and corrosion attack of the two most satisfactory blends. Rubber-swelling tendencies of some packings merit consideration, as shown by AMC data.

In addition to further investigation of halogenated snuffers, the development of fire-resistant viscosity-index improvers would be of considerable service in producing a desirable hydraulic fluid.

PROPRIETARY AND OTHER LESS-INFLAMMABLE HYDRAULIC FLUIDS

Several less-inflammable fluids are now in commercial production. These fluids contain aqueous solutions of ethylene glycol plus suitable additives, phosphate esters, or chloro or chlorofluoro snuffer compounds. In addition to Hydrolube U-4, two other commercially available fluids are being flight-tested (reference 19). The characteristic properties of these two fluids, RPM Nonflammable Hydraulic Fluid and Skydrol, were presented at a recent annual meeting of the Society of Automotive Engineers in Detroit (references 34 and 35).

In order to encourage and to accelerate the development of less-inflammable hydraulic fluids, the Aircraft Industries Association is sponsoring the evaluation of hydraulic fluids at the Cornell Aeronautical Laboratory (reference 36). The fluid, which is submitted by the manufacturer, is evaluated in accordance with the SAE AMS 3150A specification. A report of the results will be sent to the manufacturer and satisfactory fluids will be approved by the Aircraft Industries Association and the Air Transport Association.

Some fluids have not been discussed, for example, phosphate-ester Fluid N (reference 11) and water-base fluids, Hydrolube H-1, Hydrolube H-2, and Fluid 909. The primary object of the report was not to describe all the formulations that have been developed but to discuss the primary classes of material under investigation. A brief tabular summary of typical components, relative inflammability, and other important properties for the five main classes of less-inflammable fluids as compared with the AN-O-366 specification is provided in table XIV.

For completely satisfactory service in all current aircraft, some modification of either the present fluids or of present packings or metal parts of hydraulic systems is apparently necessary.

SUMMARY

The current status of the development of less-inflammable hydraulic fluids for aircraft may be summarized as follows:

1. From an investigation of glycols and derivatives, least inflammability was shown by certain glycols of low molecular weight and by polymeric glycols and their ethers or esters. Chain branching of the less-inflammable polymeric glycols would give needed improvement in pour point but not in viscosity index. Promising formulations have been prepared using polymeric glycol ethers plus less-inflammable thinners or constituents such as dicarboxylates or phosphate esters.

2. Water-base hydraulic fluids containing ethylene glycol and various additives have had considerable study and testing. Better low-temperature characteristics, less wear in pumps, and decreased attack on active metals are among the objectives in the continued development of this type of fluid.

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3. Silicones of proper viscosity have shown many qualities that would recommend their use for less-inflammable hydraulic fluids but difficulties, only partly resolved, have been met in sliding-friction wear for steel-to-steel and steel-to-cast-iron surfaces, shrinking and hardening of O-ring rubber packings, and maintenance of tight connections in hydraulic lines.

4. Esters, including phosphates, dicarboxylates, borates, and carbonates have been considered as base materials. Many of the esters, especially borates and carbonates, were considered undesirable because they readily hydrolyze. Marked improvement over petroleum-base fluids in inflammability tests has been shown by some phosphate esters; however, they caused undue swelling of rubber packings used in present aircraft hydraulic systems.

5. Halogenated organic compounds, particularly chloro and chlorofluoro compounds, have shown promise as snuffer constituents of less-inflammable hydraulic fluids but also have a pronounced effect on present rubber packings. Fluorocarbons have received some attention as possible base materials but many investigated thus far are liquid over too small a temperature range or are insufficiently soluble with other probable constituents of hydraulic fluids. Branching of the molecular structure in certain fluorocarbons has been recently suggested as a possible means to alleviate these difficulties and improve viscosity characteristics.

Several less-inflammable hydraulic fluids are now in commercial production that use the more favorable types of material named, such as aqueous solutions of ethylene glycol plus suitable additives, phosphate esters, and chloro or chlorofluoro snuffer compounds. The interest of commercial airlines and the armed services has hastened the testing of the announced manufactured fluids. Hydrolube U-4, Skydrol, and RPM Nonflammable Hydraulic Fluid have been or are being flight-tested. For completely satisfactory service, some modification of the fluids or of parts of the present hydraulic system still appears necessary.

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TABLE I - COMPARISON OF HYDRAULIC-FLUID SPECIFICATIONS

	AN-0-366 (supersedes AN-VV-0-366)	SAE AMS 3150A	Proposed military specification (Dec. 12, 1949)
Effect on present packings	Swelling of rubber L within limits established by high-and low-reference fluids	No deleterious effect	No deleterious effect ^a
Compatibility with AN-0-366 fluid	-----	Compatible with 5 percent	Compatible with 10 percent from 160° to -65° F
Toxicity	-----	Nontoxic	Require no special handling ^b
Viscosity at °F -65 (max.) -40 (max.) 150 (min.)	500 centistokes 10 centistokes	52000 centistokes 67000 centistokes	2500 centipoises 90 centipoises
Pour point (max.), °F	-75	-----	-----
Low-temperature stability	Uniform liquid at -85° F for 72 hrs	Uniform liquid at -60° to 160° F for 168 hrs	Fluid and fluid plus 10 percent AN-0-366 remain uniform liquids at -65° F for 72 hrs
Specific gravity at 60° F/60° F	Base stock determined not controlled	1.1	Determined not controlled
Corrosion	Max. weight change after 168 hrs at 250° F for steel, Al alloy, Mg alloy, Cd-plated steel, 0.2 mg/sq cm; for Cu, 0.6 mg/sq cm	Max. weight change after 168 hrs at 180° F for steel, Al alloy, Mg alloy, Cd-plated steel, 0.2 mg/sq cm; for Cu, 0.6 mg/sq cm	Max. weight change after 168 hrs at 250° F for steel, Al alloy, Mg alloy, Cd-plated steel, 0.2 mg/sq cm; for Cu, 0.2 mg/sq cm
Oxidation (of fluid during corrosion test)	Max. viscosity change at 150° F, -5 to 20 percent; max. neutralization-number increase, 0.2	Max. viscosity change at 150° F, -5 to 10 percent; max. neutralization-number increase, 0.2	Max. viscosity change at 150° F, -5 to 20 percent; max. neutralization-number increase, 0.5
Hydrolytic stability	-----	-----	Max. weight change of Cu strip after 48 hrs at 200° F in fluid plus 25-percent water, 0.5 mg/sq cm; max. neutralization-number increase, 0.5
Evaporation and tackiness	Oily but not tacky residue	Residue not tacky	Oily but not tacky residue



Shear stability	After 5000 cycles at 1000 lb/sq in., 1740 rpm, and 100° F, viscosity percentage decrease at 130° F and -40° F not greater than for AN-F-53 reference fluid; max. neutralization-number increase, 0.2	At 3000 lb/sq in., 5000 to 5600 rpm, and 160° F, equal or better performance than with AN-VV-O-368b fluid; viscosity, neutralization number, and pH determined	After 5000 cycles at 1000 lb/sq in., 1740 rpm, and 100° F, viscosity percentage decrease at 130° F must be no greater than for AN-F-53 reference fluid; max. neutralization-number increase, 0.5
Wear and lubrication	-----	Wear of Vickers PF-3811-25 pump in previous test equal to or less than with AN-VV-O-368b fluid	Wear of Pesco 549 gear pump after 600 hrs at 5600 rpm, 1500 lb/sq in., and 160° F no greater than with AN-O-366 fluid. Satisfactory operation in other pumps as determined
Volatility	-----	-----	Fluid taken from Pesco pump test must pass high-temperature ignition test
Power system	-----	At strut operation rate of 2 cycles/min at 3000 lb/sq in. and 160° F, performance at least equal to AN-VV-O-368b fluid	-----
Inflammability tests	200	750	-----
Flash point (min.), °F	-----	-----	-----
Autogenous-ignition temperature (min.), °F	-----	-----	-----
Wick	-----	-----	-----
Spray	-----	-----	-----
High-temperature ignition	-----	Performance at least equal to HS-1 reference fluid	No ignition; or self-extinguishing upon removal of test flame
Manifold (at 1300° F)	-----	-----	No ignition on manifold or upon leaving manifold
Incendiary gunfire	-----	-----	No ignition by 0.30-caliber incendiary bullet

*Fluids unsuited for present packings will be considered if manufacturer develops suitable packings, which meet functional requirements of packing specifications.

^bFluids requiring special handling because of toxicity will be considered if manufacturer furnishes toxicity data by medical authorities.

^cPreferred upper limit; satisfactory for all commercial aircraft (grade 8000).

^dMaximum upper limit; may be restricted to certain types of aircraft (grade 7000).

^eFluids having lower viscosity than allowed at 130° F will be considered if satisfactory pump life and no excessive leakage have been demonstrated.

^fHigher values acceptable if proper system performance can be demonstrated.

TABLE II - FLAMMABILITY OF GLYCOLS AND AQUEOUS MIXTURES

[Reference 7, table VI, p. 1613]

Fluid	Viscosity, Cs. at 100° F.	Flash Point ^a (C.O.C.), °F.	S.I.T. °F.	Spray Flamma- bility Limit, % Oxygen	Incendiary Fire Test, Flame Height, Feet
Ethylene glycol	8.7	240	856	40	3-8
Plus 10% water	6.9	---	862	--	---
Plus 15% water	6.0	---	---	--	3
Plus 20% water	5.1	---	871	48	---
Plus 25% water	4.4	---	---	--	1
Plus 30% water	3.8	---	880	--	---
Plus 35% water	3.2	---	885	67	2
Plus 40% water	2.8	---	892	--	---
Plus 50% water	2.2	---	903	>80	---
Plus 60% water	1.6	---	918	--	---
Plus 70% water	1.3	---	934	--	---
Plus 80% water	1.1	---	956	--	---
Diethylene glycol	16.0	280(300)	900	42	---
Plus 20% water	---	---	---	50	---
Plus 35% water	---	---	---	>80	---
Propylene glycol	19.6	230(235)	835	38	---
Dimethoxytetraethylene glycol	2.5	285 ^b	---	12	---
Plus 45% water	---	---	---	>80	---
Diethylene glycol monoethyl ether	2.8	210 ^b	790	12	---
Plus 45% water	---	---	---	60	---
Ethylene glycol monomethyl ether	1.3	115	---	12	---
Plus 45% water	---	---	---	29	---
Plus 45% water plus 25% glycol	---	---	---	>80	---
Ethylene glycol monoethyl ether	2.2 at 68°	135 ^b	---	12	---
Plus 45% water	---	---	---	51	---
Plus 45% water plus 25% glycol	---	---	---	>80	---
Ethylene glycol monobutyl ether	2.4	165 ^b	---	12	---
Plus 45% water	---	---	---	56	---
Plus 45% water plus 25% glycol	---	---	---	>80	---

^aWhen available, fire point is given in parentheses.^bManufacturer's data.

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TABLE III - COMPOSITION AND PROPERTIES OF HYDROLUBES

(a) Composition



Hydrolube A ^a	
Components	Percent by weight
Acrysol G-3493-B polymer ^b	4.8
Ethylene glycol	52.6
Water (distilled)	38.8
Triethanolamine	1.6
Phosphoric acid	.4
Diisopropylamine nitrite	1.6
Sodium mercaptobenzothiazole	.2
Hydrolube U ^a	
Ucon 75-H-69400 polymer ^b	10.2
Ethylene glycol	49.7
Water (distilled)	36.4
Diethylethanolamine	1.5
Phosphoric acid	.5
Diisopropylamine nitrite	1.6
Sodium mercaptobenzothiazole	.1
Hydrolube U-4 ^c	
Ethylene glycol	38.4
Ucon lubricant 75-H-90000 ^b	9.6
Distilled water	36.9
Ethylene glycol monobutyl ether	10.0
2-Methyl-2,4-pentanediol	2.0
Diamylammonium laurate	1.0
Diisopropylammonium nitrite	1.7
Salicylal ethanolamine	.2
Sodium mercaptobenzothiazole	.2

(b) Properties

Hydrolube	Viscosity (centistokes)				Freezing point (°F)	pH
	-40° F	0° F	100° F	130° F		
A ^a	1260	180	15	9.6	Below -60	8.0-8.2
U ^a	1760	240	16	10	Below -50	8.8-8.9
U-4	1850	245	17	10 (min.)	Below -50	8.2-8.7

^aReference 9, p. 11.^bViscosity-index improver.^cComposition for a recent commercial sample of Carbide and Carbon Chemicals Corporation.^dReference 11, table II.^eNavy specification 51F22 (Aer.), Oct., 1947.

TABLE IV - GEAR-PUMP RUNS ON DIMETHYL-SILICONE-POLYMER FLUID

[Reference 20, table 2, p. 364]



	Run No.									AN-VV-O-366b	
	P-11	P-13	P-18	P-19	P-20	P-21	P-22	P-23	P-25	P-60	P-63
Time in hours	48	50	41	100	100	100	100	100	500	100	100
Sump temp. °F	180	180	180	180	180	180	200	200	140	180	180
PSI high	1500	1500	1500	1000	1500	1500	1500	1500	1500	1500	1500
PSI low	135	110	150	150	175	140-155	160	165-195	210-300	38-40	37-45
Flow GPM:											
Start	3.24	3.25	3.35	3.24	3.34	3.35	3.24	3.36	3.51	3.08	2.97
Finish	3.27	3.35	3.24	3.24	3.34	3.34	3.12	3.35	3.43	3.04	2.92
Cycles approx.	9,360	9,750	8,118	19,440	20,040	20,040	19,080	20,100	105,000	18,000	16,000
Wt. loss grams:											
Drive gear	.0066	-----	.0117	.0034	.0048	.0006	.0092	.0044	.0030	.0439	.0536
Driven gear	.0045	-----	.0072	.0000	.0020	.0011	.0041	.0000	.0028	.0372	.0592
Max. one bushing	.0034	-----	.0068	.0081	.0010	.0010	.0086	+.0004	.0168	.1782	.0102
Total bushings	.0042	-----	.0133	.0171	.0024	.0020	.0137	+.0010	.0208	.4452	.0204
Visc. cs.:											
Start	-----	69.35	69.27	69.27	69.27	71.11	69.27	71.11	71.11	13.37	13.37
Finish	-----	68.51	68.25	68.58	69.23	72.05	70.39	72.34	70.16	7.884	8.485
Filter type	Skinner no bag	Purolator line type	Skinner with bag	Skinner with bag	Skinner no bag*	Skinner with bag	Skinner no bag**	Skinner with bag	Skinner no bag	Purolator line type	Purolator line type
Appearance of oil after run	Cloudy gel	Light amber, clear	Hazy	Hazy	Hazy	Clear, light amber	Hazy amber	Clear amber	Clear deep yellow	-----	-----
Gears & bushings	New	New	New	New	From P-19	From P-19 and P-20	New	New	New	New	Broken in 100 hrs.

*After 40 hours.

**After 60 hours.

TABLE V - VISCOSITY STABILITY AND WEAR CHARACTERISTICS OF HYDRAULIC FLUIDS CONTAINING TRI-N-BUTYL PHOSPHATE

Test Conditions: Pump = Perco 349 Gear Pump Speed = 3600 r.p.m.
 Valve = Vickers Model C-167-G Temperature = 100° F. to 200° F.
 Pressure = 1000 to 2500 p.s.i. Test Time = 100 hours (total)

Description of Fluid: 12.2 weight % Acryloid 865 in an 80-20 weight ratio mixture of tri-n-butyl phosphate and 2-ethylhexyl sebacate (PRL 2476).

[Reference 25, table XXIX, p. 132]



RUN NO.	TEST TIME, HOURS	PRESSURE, P.S.I.	OIL TEMP., °F.	FLOW RATE, G.P.M.		LOSS IN WEIGHT OF PUMP PARTS AFTER TEST, GRAMS.						% DECREASE IN 100° F. C' STOKES VISC. AFTER 5000 CYCLES	NEUT. NO. OF RESIDUE MG. KOH/GM. OIL
				INITIAL	FINAL	DRIVE GEAR	IDLER GEAR	BRONZE BUSHINGS					
								A	B	C	D		
PRL Pump Test Unit No. 3 - Vickers Model C-167-G Valve													
162	100	1000	100	3.57	3.57	0.178	0.078	0.011	0.011	0.013	0.011	28.0	0.73
163	0 to 22	1000	100	3.60	3.60	-	-	-	-	-	-	-	-
	22 to 46	2000	150	3.60	3.54	-	-	-	-	-	-	-	-
	46 to 97	2500	200	3.54	3.40	0.493	0.208	0.022	0.041	0.023	0.066	-	1.04
165	100	1000	100	3.57	3.59	0.130	0.072	0.009	0.012	0.013	0.012	26.4	0.44
Typical Hydrocarbon-Base Specification O.S. 2943 Fluid (PRL 1965)													
130	100	1000	100	3.43	3.44	0.002	0.002	0.004	0.001	0.001	0.004	-	0.2 to 0.3

TABLE VI - COMPOSITION AND PROPERTIES OF VARIOUS LESS INFLAMMABLE HYDRAULIC FLUID FORMULATIONS



[Reference 29, table 9, p. 30]

Blend Designation	PRL 2737	PRL 2738	PRL 2739	PRL 2740	PRL 2741	PRL 2742	PRL 2810	PRL 2811	PRL 2812	PRL 2743	PRL 2744	PRL 2745	PRL 2746
Composition, wt. %:													
Acryloid 855 (active) ⁽¹⁾	6.9	6.8	4.0	9.2	8.2	5.0	7.0	7.4	7.4	2.0	3.2	4.5	6.2
XCT White 011 ⁽¹⁾	5.5	21.4	3.2	27.9	4.9	3.9	5.5	5.8	5.8	1.6	2.6	3.6	4.9
Voltesso-36 ⁽¹⁾	-	20.5	-	18.8	-	-	13.1	11.8	11.8	-	-	-	-
2-Ethylhexyl Sebacate	-	-	46.4	-	44.5	45.6	-	-	-	48.2	46.7	46.0	-
Hexachlorobutadiene	87.6	51.3	46.4	-	-	22.7	74.4	50.0	25.0	-	23.9	-	47.1
Monochlor Xylene	-	-	-	-	-	-	-	-	-	-	-	-	-
Hexafluoride	-	-	-	44.0	44.4	22.8	-	25.0	25.0	-	-	23.0	-
Dichlor Xylene	-	-	-	-	-	-	-	-	-	-	-	-	-
Hexafluoride	-	-	-	-	-	-	-	-	25.0	-	-	-	-
Fluorolube FS	-	-	-	-	-	-	-	-	-	48.2	23.6	22.9	41.8
Properties:													
Viscosity in centistokes at,													
210° F.	6.04	5.71	4.77	5.76	5.20	4.86	4.9	4.8	4.4	3.66	4.39	4.67	5.44
130° F. (extrapolated)	10.7	10.9	9.9	11.0	10.7	10.1	10.1	10.1	9.0	8.8	9.8	10.2	10.3
100° F.	14.5	15.5	14.9	15.6	15.9	14.9	14.3	14.4	12.9	14.3	15.2	15.7	14.8
0° F. (extrapolated)	75	106	148	101	149	142	84	80	80	-(2)	198	190	-(2)
-25° F. (extrapolated)	157	255	434	240	430	400	170	183	164	-	480	480	-
-40° F.	285	510	971	454	989	939	268	298	253	-	1596 (3)	1594 (3)	-
A.S.T.M. slope (210° to -40° F)	0.46	0.51	0.59	0.81	0.58	0.59	0.49	0.51	0.53	0.66 (4)	0.64	0.68	0.45 (4)
A.S.T.M. pour point	<-75	<-75	<-75	<-75	<-75	<-75	<-75	<-75	<-75	<-75 (5)	<-75 (5)	<-75 (5)	<-75 (5)

(1) Typical spec. AN-0-366 hydraulic-fluid constituents.

(2) All components are not mutually soluble below 0° F.

(3) Fluids exhibit clouds at -40° F.

(4) A.S.T.M. slope for the interval of 210° to 100° F.

(5) Phase separation noted although bulk of fluid flows at -75° F.

TABLE VII - FORMULATION DATA OF SIX EXPERIMENTAL LESS
FLAMMABLE HYDRAULIC FLUIDS

[Reference 28, table XVIII, p. 45]



Components	MLO Number Percentage Expressed as % by Weight					
	3539	3541	3572	3573	3574	3606
Petroleum Base Stock	51.0	51.1	51.9	51.0	51.6	52.8
Acryloid HF-855	7.7	7.9	6.96	6.8	10.0	6.3
Paranox 441	0.4	0.4*	0.4	0.4	0.4	0.4
Fluorolube Fore Shot	15.6	17.9	16.7	16.4	15.0	7.9
Tetrachlorotetrafluoropropane	25.8	----	----	----	----	----
Chlorobis(trifluoromethyl)- benzene	----	23.1	----	----	----	----
Dichloro(trifluoromethyl)- benzene	----	----	24.	----	----	----
Dichlorobis(trifluoromethyl)- benzene	----	----	----	25.4	----	----
3,3 Difluorotetrachloropro- pene	----	----	----	----	23.	----
Chloro-1-(trifluoromethyl)-4- (chlorotetrafluoroethyl)- benzene	----	----	----	----	----	32.6

* Paranox 441 percentage not figured into the final component percentage.

TABLE VIII - PROPERTIES OF HALOGENATED SNUFFERS

[Data taken from reference 28. All temperatures have been converted from °C.]



Compounds	Viscosity at 100° F (centipoises)	Boiling range (°F)	Freezing range (°F)	Flash point (°F)	Fire point (°F)	Spontaneous ignition temperature (°F)	Cool flame temperature (°F)
3-Chloro(trifluoromethyl)benzene	0.715	275	-98.3	127.4	158	1440	1296
4-Chloro(trifluoromethyl)benzene	.708	276.8	-5.4	113	140	1450	1303
2-Chloro(trifluoromethyl)benzene	.927	300.2	24.8	69.8	129.2	1425	1303
Dichloro(trifluoromethyl)benzene	1.24	347 to 356	24.8 to 41.0	339.8	339.8	1425	1202
Chlorobis(trifluoromethyl)benzene	.967	289.4 to 293	-139 to -148	293	293	1386	1184
Dichlorobis(trifluoromethyl)benzene	1.58	334.4 to 336.2	-40 to -54.4	338	338	1483	1303
Perfluoromethylnaphthalene	4.15	321.8 to 323.6	-112	320	320	----	1321
4-(Trifluoromethyl)-1-(chlorotetrafluoroethyl)benzene	1.50	318.2	10.4 to -4	320	320	---	1206
Hexafluoroxylenes	.880	241.7	-31 to -45.4	187	230	1436	1436
Perfluorodiethylocyclohexane	1.458	291.2	-104.8	293	293	1112	1094
Tetrachlorotetrafluoropropane	1.179	235.4	-166	233.6	233.6	1382	1382
3,3-Difluorotetrachloropropene	.867	264.2	-144.4	258.8	258.8	1355	1130
Trifluoromethyloxybis(trifluoromethyl)benzene	*1.01	275.4	-121	275	275	1526	1341
Chloro-1-(trifluoromethyl)-4-(chlorotetrafluoroethyl)benzene	-----	365	-94	-----	-----	-----	-----

*At 103.8° F.

TABLE IX - HYDRAULIC FLUID INFLAMMABILITY TESTS

Tests conducted in accordance with the proposed specification for non-flammable type hydraulic fluid issued by the Aircraft Industries Association of America, Inc. and dated July 21, 1947.

[Reference 29, table 12, p. 41]



Fluid (Proportions by weight)	Wick test				Spray test	High temperature ignition test	Exhaust manifold test
	Wick	Min. time to ignite (sec.)	Max. duration of burning (sec.)	Self extinguishing			
HS-1 ⁽³⁾	Paper Fiberglas	1-1/2 1	17 13	No Yes	No increase	Flashes only	Burns on pipe only
AN-VV-0-366b (LO 2286)	Paper	1/2	14	No	Increases	Sustained fire	Burns on pipe and pan
	Fiberglas	1/2	8	No			
LO 2738 ⁽¹⁾	Paper	1	14	No	No increase	Flashes only	Burns on pipe only
	Fiberglas	1	12	No			
LO 2739 ⁽²⁾	Paper	3	15	No	No increase	Flashes only	Burns on pipe only
	Fiberglas	1	11	No			
Hexachlorobutadiene	Paper	1	21	No	Decreases	Could not be ignited	No flash on pipe or pan
	Fiberglas	No burning	Several momentary flashes				

(1) LO 2738 = 6.8 wt. % Acryloid 855 + 21.3 wt. % XCT White Oil (C.O.C. flash point, 220° F.) + 20.4 wt. % Voltesso 36 (C.O.C. flash point, 300° F.) + 51.1 wt. % hexachlorobutadiene.

(2) LO 2739 = 4.0 wt. % Acryloid 855 + 3.2 wt. % XCT White Oil (C.O.C. flash point, 220° F.) + 46.2 wt. % 2-ethylhexyl sebacate (C.O.C. flash point, 430° F.) + 46.2 wt. % hexachlorobutadiene

(3) Data for HS-1 is taken from A.I.A. proposed specification for non-flammable hydraulic fluids

Table X - FLAMMABILITY TESTS
 [Reference 28, table XX, p. 52]



Flammability Tests	AN-O-366	Experimental Less Flammable Aircraft Hydraulic Fluids					
		MLO 3539	MLO 3541	MLO 3572	MLO 3573	MLO 3574	MLO 3606
Paper Wick {	Time to ignite	0.6 sec.	1 sec.	1 sec.	These tests not performed.		
	Length of burning	15 sec.	15 sec.	15 sec.	These tests not performed.		
Fiberglass Wick {	Time to ignite	0.5 sec.	7 sec.	0.5 sec.	These tests not performed.		
	Length of burning	8 sec.	7 sec.	6.1 sec.	These tests not performed.		
Spontaneous Ignition Temperature, °F		978	1148	1150	These tests not performed.		
Spray Test		Increased Fire	----- Extinguishes Fire -----				
High Temperature Ignition Test		Vigorous Fire	----- Burns only in Oxy-Acetylene Flame -----				
Exhaust Manifold		Burns on Pan	----- No flashing or burning on manifold or in pan -----				

TABLE XI - OXIDATION AND CORROSION CHARACTERISTICS OF HALOGEN-CONTAINING COMPOUNDS AND "SNUFFER"-CONTAINING HYDRAULIC FLUIDS

[Reference 29, table 14, p. 55-57]



NACA RM E50F29

Column No. Inhibitor used Inhibitor concentration, wt. % Test fluid	AN-0-366	1 None Hexa- chloro- butadiene	2 None Fluorolube FS	3 None Monochlor xylene hexafluoride	4 Paranox 0.4 PRL 2738	5 441 0.4 PRL 2738
Overall liquid loss, wt. %	8 max.	3	3	72	4	4
% change in viscosity At 130° F. At 0° F.	-5 to 20	+1 0	+8 +25	+53 +67	-15 +55	+9 +16
Neut. No. (mg. KOH/gm. oil): Original Final	0.2 max. 0.2 max. increase	0.1 0.1	1.8 1.8	0.0 1.0	0.2 6.4	0.2 1.4
A.S.T.M. union color: Original Final	1-	1- 1-1/2	1- 1-	1- 2-1/2	1-1/2 >8	2- >8
Wt. % insoluble material	None	None	None	None	None	None
Final catalyst condition: Appearance: Copper Steel Aluminum Magnesium Cd-pltd. steel	No etching, pitting, or visible corrosion on all metals	Coated Rusted Corroded Corroded Bright	Dull Bright Bright Bright Bright	Corroded Rusted Corroded Corroded Coated	Coated Rusted Corroded Corroded Dull	Coated Rusted Corroded Corroded Bright
Wt. loss (mg./sq. cm.): Copper Steel Aluminum Magnesium Cd-pltd. steel	+0.6 +0.2 +0.2 +0.2 +0.2	+0.40 +0.06 -0.02 -0.43 -0.02	+0.05 +0.01 +0.01 +0.03 +0.01	-0.51 +0.12 +0.11 +0.11 +0.14	+0.52 +0.17 +0.02 +0.06 +0.02	+0.41 -0.25 +0.17 -0.27 +0.04

TABLE XI - OXIDATION AND CORROSION CHARACTERISTICS OF HALOGEN-CONTAINING COMPOUNDS AND "SNUFFER"-CONTAINING HYDRAULIC FLUIDS - Continued

[Reference 29, table 14, p. 55-57]



Column No.	6	7	8	9	10	11	12
Inhibitor used		Stan Add 43B		Methyl	H1 #4	B Naphthol	
Inhibitor concentration, wt. %	0.4	0.4	0.4	0.4	0.4	0.4	0.4
Test fluid	PRL 2738	PRL 2738	PRL 2739	PRL 2738	PRL 2739	PRL 2738	PRL 2739
Overall liquid loss, wt. %	5	4	3	5	4	5	14
% change in viscosity							
At 130° F.	+18	+19	+13	+4	+9	-12	+34
At 0° F.	+25	+32	+16	+159	+15	+67	+61
Neut. No. (mg. KOH/gm. oil):							
Original	0.2	0.2	0.2	0.2	0.3	0.1	0.1
Final	0.0(1)	0.5(1)	0.0(1)	0.4(1)	0.0(1)	0.4(1)	0.1(1)
A.S.T.M. union color:							
Original	1-1/2	3-1/2	2-1/2	1-1/2	2-1/2	1-1/2	2-1/2
Final	>8	>8	>8	8+	>8	>8	>8
Wt. % insoluble material	None	None	0.1	4.3	1.2	0.2	0.5
Final catalyst condition:							
Appearance:							
Copper	Bright	Coated	Coated	Dull	Corroded	Coated	Etched
Steel	Rusted	Rusted	Rusted	Lacquered	Rusted	Dull	Rusted
Aluminum	Bright	Bright	Dull	Bright	Corroded	Bright	Pitted
Magnesium	Dull	Dull	Corroded	Corroded	Corroded	Corroded	Corroded
Cd-pltd. steel	Bright	Lacquered	Lacquered	Bright	Coated	Bright	Dull
Wt. loss (mg./sq. cm.):							
Copper	-0.47	+2.50	+3.07	-0.77	-0.30	+1.02	-0.73
Steel	+0.33	+0.36	+0.74	-0.19	-0.15	-0.08	-0.51
Aluminum	0.00	+0.05	+0.03	0.00	+0.18	0.00	+0.14
Magnesium	+0.09	+0.11	+1.33	+0.67	-0.59	+0.50	-0.36
Cd-pltd. steel	-0.27	+0.64	+0.12	-0.88	+0.05	-0.04	+0.11

TABLE XI - OXIDATION AND CORROSION CHARACTERISTICS OF HALOGEN-CONTAINING COMPOUNDS AND
"SNUFFER"-CONTAINING HYDRAULIC FLUIDS - Continued

[Reference 29, table 14, p. 55-57]



Column No. Inhibitor used Inhibitor concentration, wt. %	13 Aerolube 76 0.4	14 76 0.4	15 Lubrizol 728 0.4	16 728 0.4	17 Caloco MB 0.4	18 0.4 wt.% Aero- lube 76 + 0.05 wt.% Dupont metal deacti- vator PRL 2738	19 0.05 wt.% Para- nox 512 + 0.2 wt.% mercapto- benzothiazole PRL 2738
Test fluid	PRL 2738	PRL 2738	PRL 2738	PRL 2738	PRL 2738	PRL 2738	PRL 2738
Overall liquid loss, wt. %	4	4	5	4	4	4	4
% change in viscosity At 130° F. At 0° F.	+9 +24	+10 +20	+7 +22	+26 +30	+21 +24	+7 +18	+41 +44
Neut. No. (mg.KOH/gm. oil): Original Final	0.4 0.1(1)	0.4 1.0(1)	0.5 0.1(1)	0.2 0.1(1)	0.2 0.2(1)	0.5 1.4	0.7 0.2(1)
A.S.T.M. union color: Original Final	1-1/2 >8	2-1/2 >8	3-1/2 >8	3-1/2 >8	3-1/2 >8	2- >8	4 >8
Wt. % insoluble material	None	0.1	None	0.1	None	0.1	0.1
Final catalyst condition: Appearance:							
Copper	Corroded	Etched	Corroded	Etched	Coated	Corroded	Coated
Steel	Dull	Rusted	Dull	Rusted	Rusted	Dull	Rusted
Aluminum	Bright	Pitted	Bright	Corroded	Sl. corroded	Bright	Bright
Magnesium	Corroded	Corroded	Corroded	Corroded	Bright	Bright	Dull
Cd-pltd. steel	Bright	Dull	Dull	Dull	Lacquered	Bright	Lacquered
Wt. loss (mg./sq. cm.):							
Copper	-2.30	-3.53	-3.53	-2.05	+2.32	-0.43	+2.56
Steel	-0.05	+0.20	+0.07	+0.05	+0.32	+0.03	+0.46
Aluminum	0.00	+0.06	0.00	+0.11	+0.05	0.00	+0.05
Magnesium	+0.33	-0.31	+1.15	-2.18	+0.09	+0.01	+0.11
Cd-pltd. steel	-0.02	-0.08	+0.02	+0.02	+0.55	-0.05	+0.75

TABLE XI - OXIDATION AND CORROSION CHARACTERISTICS OF HALOGEN-CONTAINING COMPOUNDS AND
"SNUFFER"-CONTAINING HYDRAULIC FLUIDS - Continued

[Reference 29, table 14, p. 55-57]



Column No. Inhibitor used	20 0.4 wt.% Para- nox 441 + 0.05 wt.% Dupont metal deacti- vator	21 0.4 wt.% Para- nox 441 + 0.05 wt.% Dupont metal deacti- vator	22 0.4 wt.% Stan- Add 43B + 0.05 wt.% Paranox 512 + 0.05 wt. % Dupont metal deactivator PRL 2738	23 2.6 wt.% (2) Paranox 447A	24 2.6 wt.% Para- nox 447A + 0.2 wt.% mercapto- benzothiazole	25 2.6 wt.% Para- nox 447A + 0.05 wt.% Dupont metal deacti- vator
Test fluid	PRL 2738	PRL 2738	PRL 2738	PRL 2738	PRL 2738	PRL 2738
Overall liquid loss, wt. %	3	4	3	4	4	4
% change in viscosity At 130° F. At 0° F.	-5 +72	-6 +65	+20 +20	+9 +13	+21 +24	+9 +20
Neut. No. (mg. KOH/gm. oil): Original Final	0.2 8.6	0.2 8.4	0.3 0.3 (1)	0.2 0.2	0.7 0.2 (1)	0.3 0.4 (1)
A.S.T.M. union color: Original Final	2- >8	2- >8	2-1/2 >8	1-1/2 7-	2-1/2 >8	2- >8
Wt. % insoluble material	None	None	None	0.2	None	None
Final catalyst condition: Appearance: Copper Steel Aluminum Magnesium Cd-pltd. steel	Coated Dull Bright Dull Coated	Coated Dull Bright Dull Coated	Coated Rusted Bright Dull Lacquered	Coated Sl. rusted Corroded Sl. corroded Corroded	Corroded Sl. rusted Bright Bright Lacquered	Sl. coated Sl. rusted Sl. corroded Sl. corroded Bright
Wt. loss (mg./sq. cm.): Copper Steel Aluminum Magnesium Cd-pltd. steel	0.00 +0.03 +0.05 +0.13 +0.50	+0.08 +0.01 +0.03 +0.14 +0.34	+1.02 +0.67 +0.05 +0.13 +0.94	+0.21 +0.09 +0.18 +0.10 -3.00	-3.65 +0.05 +0.05 +0.06 +0.08	+0.16 +0.08 +0.12 +0.07 -0.06

TABLE XI - OXIDATION AND CORROSION CHARACTERISTICS OF HALOGEN-CONTAINING COMPOUNDS AND "SNUFFER"-CONTAINING HYDRAULIC FLUIDS - Concluded

[Reference 29, table 14, p. 55-57]



Column No. Inhibitor used	26 0.4 wt.% Para- nox 441 + 0.05 wt.% Paranox 512 + 0.05 wt. % Dupont metal deactivator PRL 2738	27 0.4 wt.% Para- nox 441 + 0.05 wt.% Paranox 512 + 0.05 wt. % Dupont metal deactivator PRL 2738	28 0.4 wt.% Paranox 441 + 0.05 wt.% Paranox 512 + 0.05 wt.% Dupont metal deactivator + 0.4 wt.% tetrabutyl tin PRL 2738
Test fluid	PRL 2738	PRL 2738	PRL 2738
Overall liquid loss, wt. %	3	3	2
% change in viscosity At 130° F. At 0° F.	+10 +20	+9 +20	+6 +12
Neut. No. (mg.KOH/gm. oil): Original Final	0.4 1.0	0.4 1.0	0.3 0.6
A.S.T.M. union color: Original Final	2- >8	2- >8	-- 4
Wt. % insoluble material	None	0.1	None
Final catalyst condition: Appearance:			
Copper	Dull	Dull	Dull
Steel	Dull	Dull	Bright
Aluminum	Sl. corroded	Sl. corroded	Bright
Magnesium	Dull	Dull	Bright
Cd-pltd. steel	Bright	Dull	Bright
Wt. loss (mg./sq. cm.):			
Copper	+0.15	+0.16	+0.09
Steel	+0.01	0.00	+0.10
Aluminum	+0.03	+0.03	+0.12
Magnesium	+0.05	+0.06	+0.09
Cd-pltd. steel	+0.08	+0.01	+0.07

(1) Fluorescein used as indicator instead of bromthymolblue.

(2) Paranox 447A = 16 wt. % Paranox 441 + 2 wt. % Paranox 512 in Voltesso 36.

TABLE XII - TEST DATA OF LESS FLAMMABLE FLUIDS AS COMPARED TO SPECIFICATION AN-O-366 REQUIREMENTS

[Reference 28, table XIX, p. 48]



Specification AN-O-366 Tests	Requirements	Experimental Less Flammable Aircraft Hydraulic Fluids					
		MLO 3539	MLO 3541	MLO 3572	MLO 3573	MLO 3574	MLO 3606
Neutralization No.	0.20	0.51	0.25	0.32	0.59	0.22	0.29
Color	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Viscosity (Centistokes)							
• -40° F	500	409	404	335	428	422	445
• 150° F	10	10.8	9.38	8.4	8.6	12.3	8.5
Viscosity (Centipoises)							
• -40° F	440	467	441	378	492	486	500
• 150° F	8.76	11.4	10.0	8.75	9.1	13.1	8.9
Fire Point, Open Cup, °F	240	390	390	405	390	450	340
Copper Strip Corrosion							
72 hour • 212° F	Slight brown stain	Pass	Pass	Pass	Pass	Pass	Pass
Evaporation	Shall be oily	Pass	Pass	Pass	Pass	Pass	Pass
Low Temperature Stability	-65° F	Pass	Pass	Pass	Pass	Pass	Pass
Oxidation Corrosion							
Stability • 212° F	Loss or gain						
Copper	0.2 mg/sq cm	0.08	0.128	Nil	Nil	Nil	Nil
Steel	0.2 mg/sq cm	Nil	Nil	Nil	Nil	Nil	Nil
Cadmium Steel	0.2 mg/sq cm	Nil	Nil	Nil	Nil	Nil	Nil
Aluminum	0.2 mg/sq cm	Nil	Nil	Nil	Nil	Nil	Nil
Magnesium	0.2 mg/sq cm	0.04	0.104	Nil	Nil	Nil	Nil
Visible Corrosion after							
Test							
Copper	No etching, pitting	Slight brown stain	Bright	Bright	Bright	Bright	Bright
Steel	or visible corrosion	Bright	Bright	Bright	Bright	Bright	Bright
Cadmium plated steel	on all metals	Bright	Bright	Bright	Bright	Bright	Bright
Aluminum	"	Bright	Bright	Bright	Bright	Bright	Bright
Magnesium	"	Bright	Bright	Bright	Bright	Bright	Bright
Weight % Loss of Oil							
after Oxidation-Corrosion	8% max.	5.8	5.0	6.2	6.5	1.8	3.9
Viscosity Change in %							
• 150° F	-5 to +20%	1.5	2	2	2	0	1
Neutralization No. Increase	+0.2 max.	-0.30	-0.38	-0.16	-0.19	-0.04	-0.23
Rubber Swell	% Volume increase						
Rubber F (Perbunan 26)	8.5% - 14.5%	16.8	20.2	These tests not performed.			
Rubber H (Hycar OR-25)	5.5% - 9.5%	11.6	16.3	These tests not performed.			
Rubber K (Neoprene)	33% - 52%	46.4	43.8	These tests not performed.			
Pour Point	-75	-80	-80	-75	-80	-75	-90

TABLE XIII - PUMP TESTS CONDUCTED WITH LESS-INFLAMMABLE HYDRAULIC FLUIDS IN A PESCO 349 GEAR PUMP

All Tests Conducted in a Pesco Model 349 Gear Pump Loaded by a Vickers Model 167G Pressure Relief Valve

[Reference 29, table 13, p. 52]



Test fluid	PRL 2286 (Spec. AN-VV-O-366b)		PRL 2785 (Snuffer Type Fluid)		PRL 2724 (Spec. AN-VV-O-366b) + 1% Tricresyl Phosphate	
Pump Test Conditions:						
Pressure, p.s.i.	1000	2500	1000	2500	1000	2500
Speed, r.p.m.	1800	3600	1800	3600	1800	3600
Temperature, °F.	100	200	100	200	100	200
Time, hrs.	100	100	100	100	100	100
Flow rate, g.p.m.	1.80	3.43	1.75	3.48	1.75	3.26
Loss in Wt. of Pump						
Parts, gms.:						
Drive Gear	0.008	0.104	0.004	0.061	0.001	0.026
Driven Gear	0.024	0.090	0.006	0.050	0.007	0.018
Bushing A	0.013	0.038	0.000	0.002	0.012	0.020
Bushing B	0.014	0.045	0.002	0.036	0.004	0.056
Bushing C	0.003	0.051	0.000	0.006	0.009	0.024
Bushing D	0.009	0.033	0.000	0.036	0.011	0.030
Total, 4 Bushings	0.039	0.167	0.002	0.080	0.036	0.130
Test Fluid Properties:						
Centistoke Viscosity at 100° F.	14.1	14.1	14.5	11.6 (1)	14.3	14.3
5000 Cycle Viscosity Decrease, %	21	40	22	--- (1)	20	38

(1) The charge for this test consisted of 4000 ml. of the residue from the mild condition test plus 1000 ml. of new PRL 2785 makeup fluid. Therefore, the measure of the viscosity decrease due to shear in this test is not directly comparable to the other values listed in this table.

TABLE XIV - PROPERTIES OF LESS-INFLAMMABLE FLUIDS COMPARED WITH AN-O-366 FLUID



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	AN-O-366	Glycol derivative	Water base	Silicone	Ester base	Halogenated snuffer
Typical components	Petroleum fractions, viscosity-index improver, inhibitors, and antiwear additive	Polyglycol derivative, dicarboxylate or phosphate, viscosity-index improver, and inhibitors	Water, ethylene glycol, other glycols, and vapor-phase and other inhibitors	Polymethyl or poly-(methyl, phenyl) siloxane	Phosphate (or dicarboxylate, borate, or carbonate) esters, viscosity-index improver, and inhibitors	Chloro, chloro-fluoro compounds, petroleum fractions, viscosity-index improver, and oxidation-corrosion inhibitor
Inflammability	Inflammable	Greatly decreased	Very greatly decreased unless water escapes	Greatly decreased	Decrease depending on ester type utilized	Very greatly decreased, depending on amount of suitable snuffer employed
Viscosity (centistokes) at 150° F at -40° F	10 (min.) 500 (max.)	^a 10 ^a 5500	^a 10 ^a 1850	^a 34 ^a 335	^b 27 (at 100° F) ^b 779	^c 9 ^c 404
Corrosion of metals	Noncorrosive	-----	Magnesium attacked	Noncorrosive	Noncorrosive if inhibited	Noncorrosive with selected inhibitors
Effect on standard packings	Present packing designed for this fluid	Poor, due to ester content	Satisfactory, particularly below 160° F ^d	Poor ^e	Cause swelling	Cause swelling
Toxicity	Nontoxic	-----	Nontoxic	Silicon dioxide dust from fires harmful to personnel	Nontoxic	Questionable
Specific gravity at 68° F	0.80-0.85	^a 1.06	^a 1.06	^f 0.95-0.99	-----	-----
Compatibility	-----	-----	-----	Very slight	-----	Miscible
Pump life	-----	-----	Less than AN-O-366	Less than AN-O-366 ^g	Greater than AN-O-366 ^h	About equal to AN-O-366
Relative cost per gallon	Low	Moderate	Moderate	Very high	High	High

^aReference 11.^bReference 25.^cReference 28.^dReference 37.^eReference 9.^fReference 21.^gReference 20.

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